Fuel Efficiency

Going round the works at night was like being in an enchanted land. None of your dull modern economical appliances, but a riot of escaping steam ; flames from the furnaces and sparks from the rolls and hammers dominated the scene. There were no electric lights and very little gas. The mist from the hot canals, into which condensed steam was fed, crept through the works at times and produced a most weird effect.

ONE may be sure that in making these observations on his early experiences in the iron and steel industry, during the course of what must surely be one of the most interesting Presidential Addresses ever delivered before the Iron and Steel Institute, the late W. R. Lysaght was not oblivious of the need to prevent fuel waste. The importance of fuel economy has grown in recent years with the sharp rise in the price of coal. Elsewhere in his address, Mr. Lysaght commented that, in those earlier days, " Coal, like silver in the days of Solomon, was not much accounted of," and well may we believe it when we consider that the pithead cost varied from 2s. to 5s. per ton of 2,400 lb. To-day the iron and steel industry's annual consumption of coal is of the order of 26 million tons and the bill for all forms of fuel amounts to between £70 and £80 million a year, nearly one-fifth of its total costs. Small wonder, therefore, that the industry, the largest single consumer of fuel and power in the country, is deeply concerned with the problem of ensuring that the right fuel is used for each particular operation, that it is used with the maximum efficiency and that waste is reduced to the absolute minimum.

Evidence of the industry's continued quest for fuel economy is afforded by the references to the subject in the report on its development plans, the opening paragraph of which recalls the official suggestion that priority should be given *inter alia* to schemes to reduce fuel costs and consumption. The mere saving of fuel is not to be regarded as an end in itself, for the main objective is the production of finished steel of appropriate quality at the lowest possible cost, and in the British Iron and Steel Federation's *Statistical Bulletin* for February is presented an account of the progress attained so far.

If the blast furnace, the steel plant and the rolling mill are each considered as separate manufacturing units, it is obvious that a considerable amount of heat is wasted in allowing the pig iron to solidify prior to charging into the steel furnace, in allowing ingots to go cold before heating for rolling or forging and also during the cooling of the forgings, bars, etc., after hot working. These points have, of course, long been appreciated and, with the exception of the heat loss after hot working, have been minimised in integrated steel plants, comprising coke ovens, blast furnaces, steel plant and rolling mills, in which most of the country's steel output,

particularly of unalloyed or low-alloyed structural qualities, is now made. In addition considerable savings are effected by the fullest possible use of blast-furnace and coke-oven gas. It is now generally recognised that the most economical course is to utilise blast-furnace gas, surplus to the requirements of hot-blast stoves, blowers, etc., for heating the coke ovens, leaving the much richer coke-oven gas for use in steel furnaces and reheating furnaces.

Considerable savings in fuel have also been achieved by such indirect means as pre-treatment of raw materials and modifications to plant and processes. Pig iron production absorbs some 10 million tons of coke, corresponding to 15 million tons of raw coal, and a proportion of this fuel is consumed in dealing with the gangue or earthy material associated with the ore, and lost in the molten slag tapped from the furnace. Accordingly the coke consumption on high grade ores is much smaller, per ton of iron produced, than in the case of lean home ores, and steps are now being taken to use some form of beneficiation treatment on the lean ores. Much progress has already been made in ore preparation in the physical sense—crushing, grading, blending, screening and sintering—whereby the blast furnace is enabled to work more efficiently. One large works, operating on home ore, by installing additional ore preparation plant, has achieved a saving of 150 lb. coke/ton of iron produced and also a 10% increase in output. Taken over the industry as a whole, coke consumption per ton of iron has fallen from about 26 cwt. in the early 1920's to about 20 cwt. to-day. With the new blast-furnace plants, equipped with suitable ore preparation plant, it is estimated that on completion of the development plan, the figure will be down to 18 cwt.

Owing to the scarcity of good quality producer coals, many steel furnaces, in plants where coke-oven gas is not available, have been converted to liquid fuel firing, using tar, creosote-pitch or petroleum oil. Apart from being technically more efficient the long-term upward trend of coal prices has also favoured the change ; whether the increased cost of oil resulting from devaluation will cause a reversion remains to be seen.

Apart from fuel for reheating furnaces, the main fuel consumption in the rolling mills is in the form of power, and here again, improved lubrication, the adoption of cluster mills and, above all, the development of continuous mills have all contributed to a saving of fuel.

As a result of the efforts made during the past 30 years, the coal consumption per ton of finished steel has dropped from 63 cwt. in 1924 to a little over 37 cwt. in 1949. For two reasons the figures for recent years do less than justice to the industry's achievement. The first is the change in customers' requirements in favour of such products as sheets, cold-rolled strip and bright bar, which require more work and heat treatment per ton, and the second is the sharp decline in the quality of British coal.

Some Heat-treatable Copper-rich Alloys

By P. Mabb

Although copper and its principal alloys, brass and bronze, have long figured amongst the most widely used engineering materials, it is only relatively recently that alloys whose properties are enhanced by heat treatment have come into large-scale use. A few of the more important ones, including aluminium bronze and beryllium copper, are discussed in this article.

SEVERAL factors have been responsible for the rather slow adoption by industry of the heat-treatable copper alloys. Much research work has been involved in the development of the alloys; new foundry techniques have had to be introduced so that the additions may be made under strict control and without introducing unwanted oxides or other impurities; and, perhaps the most important factor of all, the prices have been rather high when considered in relation to the extent to which the properties are improved. Nevertheless, such alloys are now finding a place in the engineering industry and, as their use becomes more widespread, their cost will tend to fall and their availability to increase.

ALUMINIUM BRONZE

Of the copper-rich alloys which respond to heat treatment, first mention may be made of the aluminium bronzes. These alloys have been known since the beginning of the century and came into considerable prominence some three decades ago. Nevertheless, in Great Britain, their adoption was slow and their use mainly restricted to certain heavy engineering applications, and to aircraft and automobile engineering; a small amount was used in chemical engineering. Although they are among the oldest heat-treatable copper alloys, it may be of interest to mention one or two points concerning their characteristics.

The commercial aluminium bronzes comprise two main groups of alloys: the α alloys contain 4–7% of aluminium and the $\alpha + \beta$ alloys 8·5–11%. The α alloys are characterised by the fact that they can be cold-worked whereas the $\alpha + \beta$ alloys are only suitable for hot working. The cold-worked α alloys, which may have a hardness of 220 D.P.N. with a corresponding tensile strength in the neighbourhood of 45 tons/in.², have many uses where a material is called for having greater resistance to abrasion and corrosion than is obtainable with the common brasses and certain of the high-tensile brasses.

The Alpha-beta Alloys

As mentioned earlier, the $\alpha + \beta$ alloys can only be fabricated by hot working—i.e., they can be produced as castings, forgings, hot pressings and extrusions, and will only allow very slight cold-working operations to produce finished dimensions. Although the range of aluminium content was given earlier as 8·5–11%, in practice it is usually 9·5–11%. The reason for the hardening of the $\alpha + \beta$ bronzes by thermal treatment lies in the fact that the $\alpha + \beta$ structure can be changed into the β condition when the material is heated to 800°–900° C. On quenching it is not possible to retain the whole of the α phase in solid solution and the typical appearance of a specimen which has been heated and quenched correctly is similar to acicular martensite with the α phase showing as white streaks. If the material in this state is then heated to a suitable temperature, the $\alpha + \beta$ structure breaks down into $\alpha + \gamma_2$. The γ_2 constituent is hard and its presence increases the hardness of the alloy and also its ultimate tensile strength.

Effect of Other Elements

The properties of the $\alpha + \beta$ aluminium bronzes are modified by the presence of other alloying elements. Iron, for instance, increases the tensile strength, but perhaps its main influence is in restricting excessive grain growth during heat treatment. Up to about 4% there is an increase in tensile strength of about 2·0–2·5 tons/in.² for each 1% of iron added. Few commercial alloys contain as much as 5%.

Nickel is beneficial in increasing the density of the cast alloy; this is of particular value in castings required for use in hydraulic engineering. It also increases the tensile strength and improves the resistance to sea-water corrosion. Nickel additions are usually of the order of 5%.

Small additions of manganese improve the mechanical properties in that the yield point and tensile strength are increased without any marked reduction in ductility. 1% of Manganese raises the yield point by 3–4 tons/in². It is also claimed that resistance to abrasion is improved and that no reduction in corrosion resistance results.

It cannot be claimed that aluminium bronze is a free machining material: the 10% alloy can only be machined at approximately the same speeds and feeds as would be employed for a steel of about 50 tons/in.² tensile strength. As in the case of brass, the addition of lead improves the machinability and in the amounts usually added does not detract from the mechanical properties except with respect to ductility and impact strength.

Lead is, however, very objectionable from the point of view of hot rolling, as it may give rise to serious cracking. For this reason leaded aluminium bronze rods and sections are produced only by extrusion. Lead is usually added to casting aluminium bronzes to improve frictional properties rather than machinability.

Heat Treatment

The composition of a typical heat-treatable aluminium bronze would be :—

Al	Fe	Pb	Ni	Zn	Sn
9·5–11%	1·5% max.	1% max.	0·075% max.	0·5% max.	Trace

Extruded rods produced from this material would have hardness values ranging from 150–220 D.P.N.

T.. material in the hot-forged state would have hardness values more closely approaching the upper figure. It will be appreciated that, this alloy being responsive to heat treatment, the structure in extruded rods will vary throughout the length, and from rod to rod. This variation will depend on the temperature of the billet at the commencement of extrusion, the gradual reduction of the temperature of the billet in the extrusion press, and the different rates of cooling to which the rod is subjected.

In order to get the best results, it is advisable, therefore, to resort to heat treatment. This, for an alloy of composition similar to that referred to above, consists of quenching from 855°–865° C., when the hardness will be around 200 D.P.N. For tempering, a suitable temperature range is 640°–660° C., the material being held at temperature for 30–45 minutes. The hardness will then lie in the range 135–175 D.P.N.

At elevated temperatures, aluminium bronze is very prone to grain growth ; it is desirable, therefore, that the lowest possible temperature for heat treatment should be adopted. For this reason, it is advisable to ensure that the aluminium content does not fall below 10%. The time at temperature should be the minimum necessary to ensure that the parts are uniformly heated throughout their mass.

Pickling

Aluminium bronze develops refractory oxide films during heat treatment and these are not so readily removed as those on brasses and the ordinary bronzes. A 5–10% sulphuric acid bath, operating at 60°–80° C., satisfactorily attacks the heaviest black oxide coat without undue damage to the base metal ; immersion times of about a minute usually suffice. Such a pickling treatment does not leave the alloy in its pleasant golden yellow state ; to achieve this it is necessary to give a further pickle in cold 5% sulphuric acid with the addition of 1% potassium dichromate. If a more brilliant appearance is desired, a rapid dip for 1–3 seconds, in a solution of 3 parts of water, 6 parts of sulphuric acid and 2 parts of nitric acid restores the maximum brilliance.

Aluminium bronze exhibits good corrosion-resisting properties, due to the formation of an inert oxide film, and is a valuable addition to the non-ferrous alloys in this respect. It is extensively used in chemical-plant engineering, offering increased life compared with a number of other alloys, and is particularly useful in the paper-making industry.

Fig. 1.—Part of the copper-beryllium equilibrium diagram.

BERYLLIUM COPPER

Beryllium copper is an age-hardening alloy, usually containing about 2% beryllium and 0·5% cobalt. It has been most widely used in the production of springs because of its exceptionally high-tensile and fatigue strengths, its hardness, its reasonable electrical conductivity and its resistance to wear and corrosion. It is also non-magnetic and largely retains its physical properties at temperatures up to 250° C. These properties are superior to those exhibited by phosphor bronze and nickel silver and, by virtue of the fact that they are obtained by heat treatment instead of cold working, the uniformity from batch to batch is greater.

In Fig. 1 the copper end of the copper-beryllium equilibrium diagram is presented. It will be seen that, at temperatures below 300° C., less than 0·2% beryllium is retained in solid solution. Normally, commercial alloys contain from 1·9–2·2% of beryllium, and it will be seen from the diagram that such an alloy, heated to 800° C., will be in the alpha condition. If the alloy is then quenched and heated to a temperature below 576° C., the a solid solution will precipitate the γ phase. Usually, the precipitation temperature employed is 300°–320° C., the time at temperature ranging from 1–4 hours, depending on the degree of cold working after the initial quench from 800° C.

In common with other copper alloys, beryllium copper has a tendency towards rapid grain growth at elevated temperatures, and it has been found that this effect can be minimised by certain addition-elements, notably nickel and cobalt. It is common for these alloys to contain 0·25–0·75% cobalt, which further tends to combine with beryllium to form a beryllite, thus reducing the amount of beryllium remaining in solution in the a phase. Provided that founding practice has been satisfactory, cobalt will also tend to retard the precipitation of the γ phase at the grain boundaries. Silicon is usually present to the extent of 0·075–0·15%.
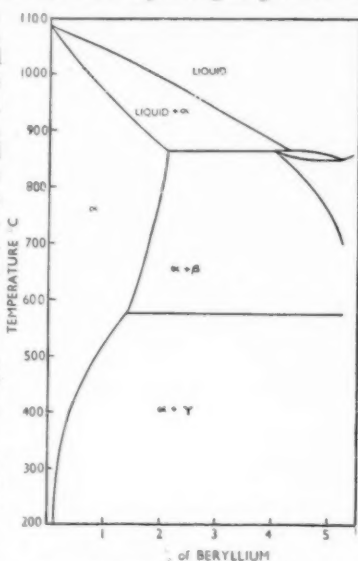
Throughout the whole of the manufacturing procedure
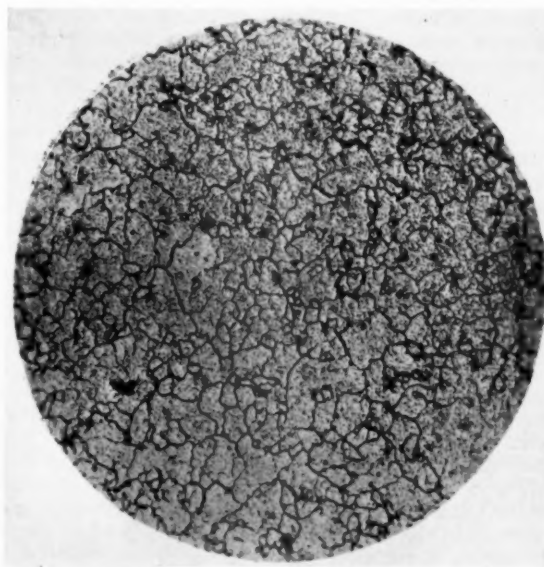

Fig. 2.—Structure of 1-in. diameter bar of beryllium copper after solution treatment and cold working to the half-hard condition. × 300

Reduced slightly in reproduction.

Fig. 3.—Structure of 0·048 in. thick beryllium copper strip, solution-treated and rolled to the half-hard condition.   × 500

Fig. 4.—Structure of beryllium copper strip after precipitation hardening by heating to 310° C. for 1½ hours.   × 500

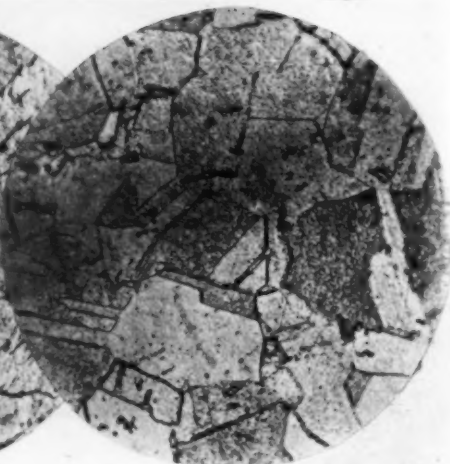Fig. 5.—Structure of the same material after solution treatment by quenching from 800° C. × 500

it is necessary to exercise effective control of the operations. Bearing in mind that the outstanding properties of the alloy depend on the beryllium and, to a lesser degree, cobalt, it is important to ensure that loss is avoided in the melting and casting stages. The high cost of these materials adds further emphasis to this point. Again, the rate of cooling of the ingots plays an important part as, if cooling throughout the mass is irregular, the β phase may be present as stringers or in massive segregates. Such ingots are difficult to correct by subsequent heat treatment or working processes. It is most important that the β phase should be uniformly distributed, and at a minimum, to ensure that there is a maximum amount of available beryllium in the α phase. Similarly in reducing the ingot, mill operations must be controlled to avoid a large amount of grain-boundary precipitate. In both cases, the effect of faulty structure will be to slow up the solution of the beryllium where segregation has occurred.

In the solution-treated condition, the alloy is relatively soft and, therefore, admirably suited for cold working and forming. Nearly all material is supplied in the solution-treated or solution-treated and cold-worked condition, particularly rod, strip and wire. It is important that the alloy should be uniformly heated throughout for the minimum length of time necessary to ensure solution and that the quenching be carried out in such a way as to retard any precipitation effects. Subsequently the material is pickled and then cold worked to varying degrees of hardness. Unless the solution-treatment is carefully carried out, a variable product results which introduces considerable trouble in producing satisfactory parts after precipitation treatment; the alloy has suffered unjust criticism on this account.

Fig. 2 shows a typical microstructure, obtained on a 1-in. diameter rod which has been solution-treated and cold-worked to an approximately half-hard condition. It will be noted that the structure is uniform with a complete absence of any β phase. Fig. 3 represents the microstructure of a sample of 0·048 in. thick strip, solution-treated and rolled to the half-hard condition.

Distortion of the grains due to cold rolling and the presence of strain effects across the grains are clearly shown.

Precipitation as mentioned above, is carried out at 300°–320° C., the higher temperature being chosen for work in the soft condition and the lower one for work-hardened material. It may also require a longer period to promote full precipitation in the soft material than in the hard-worked material. Fig. 4 shows a typical structure of strip material after precipitation hardening for 1½ hours at 310° C. It will be noted that some of the grain boundaries are broad and precipitation has occurred there. Fig. 5 is the same material, solution-treated by quenching from 800° C.

Properties

A typical specification for wrought material, in the solution-treated and worked form, would be as follows:

Composition: %
    Beryllium   ..   ..   1·9 –2·2
    Cobalt   ..   ..   0·25 –0·75
    Silicon ..   ..   0·075–0·15
    Copper ..   ..   Remainder
    Other impurities, including lead
        and silver   ..   ..   0·4 max.

Mechanical Properties:

	Hardness Range D.P.N.	Tensile Strength Tons/in.²	Elongation %
Annealed	120 max.	30–35	45–60
Quarter-hard	160–185	35–40	25–35
Half-hard	190–225	40–50	10–20
Hard	230–250	50–55	5–10

The ductility of the material is shown in Fig. 6.

Hardness after Precipitation.—The degree of hardness obtainable in beryllium copper, after precipitation hardening, ranges from 345–420 D.P.N. The hardness of the specimen shown in Fig. 5 was 97 D.P.N., that in Fig. 3, 197 D.P.N. and of the precipitation-hardened
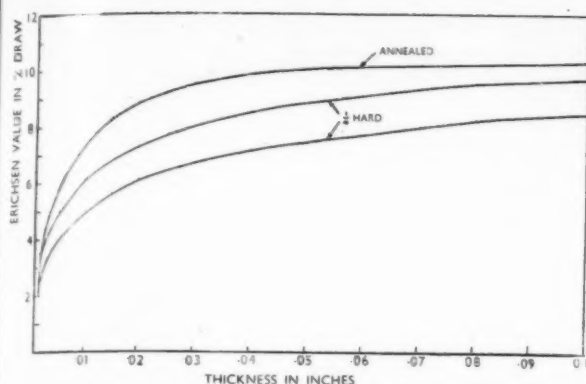
Fig. 6.—Graph showing the ductility of annealed and quarter-hard beryllium copper sheet.

specimen shown in Fig. 4, 404 D.P.N. When it is considered that phosphor bronze, in extra hard temper, usually has hardness values ranging between 220 and 240 D.P.N., the greater scope offered by beryllium copper will be obvious.

Electrical Conductivity.—The electrical conductivity of hardened beryllium copper is only 24% I.A.C.S. and any improvement which could be obtained would be an advantage. It has been found that the composition may be modified by an addition of silver to this end and an alloy of—

Beryllium	..	..	..	..	..	0·4–0·8%
Cobalt	..	..	..	..	..	1·5–2%
Silver	..	..	..	..	..	1% min.

has improved electrical properties.

Inspection

From the user's point of view, the alloy is of greatest interest in the solution-treated and cold-worked condition. Unless all stages of manufacture have been vigorously controlled material may be received which does not respond uniformly to precipitation hardening, or which is found to suffer from a number of surface defects, such as slivers, rolled-in oxide and scale, and incipient cracking along the edges resulting from the rolling operation. From a practical inspection point of view, it is usually advisable to take samples of the material from each strip or coil and subject them to precipitation treatment to ensure that consignments are uniform. A check on composition is also desirable because a shortage of beryllium or cobalt will have an adverse effect on the physical characteristics as well as reducing the maximum hardness which may be obtained.

Fabrication

In selecting material for fabrication the right degree of temper is necessary to avoid undue straining of the metal during manufacture of the parts and to ensure that no cracking troubles will arise. The alloy is available in four tempers with a useful range of hardness. It should be borne in mind that beryllium copper will work-harden at a greater rate than phosphor bronze or nickel silver and, therefore, in general, a slightly lower hardness range is desirable than in the case of the former alloys. If tool design is correct, the correct range of hardness selected, and the operation lay-out for producing a part well chosen, there is little danger of any change of form of the component after precipitation treatment.

There is always a danger, in changing over from phosphor bronze or nickel silver to beryllium copper, that trouble may be experienced when it is necessary to adjust flat-type springs. Operators who are accustomed to use considerable flexing to obtain adjustments and correct tensions may break the harder and more brittle precipitation-hardened beryllium copper. It is not easy to emphasise the difference between the two materials and to ensure that all concerned appreciate it properly. As an illustration of this point, 0·006 in. thick phosphor bronze may be bent at right angles round a 0·003 in. radius, whereas hardened beryllium copper of the same thickness requires a 0·062 in. radius if cracking is to be avoided.

Scaling During Heat Treatment

Beryllium copper has excellent corrosion resistance due to its capacity to form a protective film. Similarly, when heat treated, unless proper care is exercised, a refractory scale may form which is not easy to remove by ordinary pickling operations. It is important, therefore, that the atmosphere of the furnace used for solution treatment should be properly adjusted. A furnace with circulating heat flow is to be preferred and, if convenient, the material should be heated in a cracked ammonia atmosphere. Good results can be obtained using town's gas, but it is important that, after cracking, it should be freed from sulphur by passing through bog-ore towers. Silica gel driers should also be used to remove moisture, otherwise the alloy will scale rather badly. If scaling cannot be avoided, it is preferable to allow a black scale to be formed, rather than a very light brown one as the latter is largely beryllium oxide, is very abrasive and is only removed with difficulty.

It is not often necessary for the user to carry out solution heat treatment but, where the need does arise, every care should be taken to avoid scaling. The degree of scaling which occurs during heat treatment depends, to some extent, on the condition of the strip or rod as received. It has been noticed, for example, that flat-spring specimens, after heat treatment, although scaled on the rolled surfaces, are quite bright on the cut edges. Similarly, parts polished with abrasive paper are much brighter after quenching than if they are untouched. The same result cannot be achieved if the surfaces are pickled before treatment.

Provided that the surfaces are in good condition, little trouble is experienced with oxide effects during the precipitation treatment. This treatment is, again, best carried out in a circulating air furnace. Salt baths composed of nitrate/nitrite mixtures are also quite suitable but they should not be used for ferrous work because the latter will become coated with a browny copper deposit. After salt-bath treatment, it is essential that the parts be thoroughly washed in boiling water, cold water and hot water, to ensure removal of any remaining salt before they are passed to the stores.

Pickling

In the worst cases of scaling, the pickling treatment must be a severe one and a useful pickle is hot 10% sulphuric acid at 60°–70° C., but the subsequent surface is not very pleasing. If a brighter appearance is desired, the work should be immersed for a period of 1–2 minutes in a 5%, by volume, solution of sulphuric acid to which 2–3% sodium dichromate has been added. This solution is used at 20°–25°C. After all pickling treatments the work should be thoroughly washed in hot water and dried. In some instances, it is the practice to follow the

pickling treatments by immersion in a weak solution of sodium triphosphate, followed by water washings. The necessity for efficient pickling cannot be over-emphasised because, after solution treatment, the parts are often drawn or formed into shapes and the work is sufficiently severe to iron into the surface any retained oxide. This becomes more difficult to remove after precipitation hardening and gives rise to difficulties in electroplating.

Work pickled by sulphuric-dichromate solutions will not plate satisfactorily, the coating having poor adherence. It is, therefore, desirable to apply a bright acid dip of the sulphuric-nitric type prior to electroplating. A typical treatment is given below :—

1. Degrease in trichlorethylene.
2. Short dip in 3 parts nitric acid and 1 part sulphuric acid.
3. Water rinse.
4. Short dip in 3 parts sulphuric acid and 1 part nitric acid.
5. Water rinse.
6. Electroplate.

CHROMIUM COPPER

Copper containing small amounts of chromium has been used for many years in the manufacture of spot-welding electrodes, but more recently its use in the electrical industry has expanded because of its good combination of mechanical, electrical and thermal properties. Like beryllium copper, in the solution-treated condition it is comparatively soft and ductile and easy to fabricate by rolling, drawing or forming, etc. After precipitation hardening its tensile strength may be increased to 29–35 tons/in.^{2} (with corresponding hardness values of 142–160 D.P.N.), depending on the degree of cold working to which the material is subjected between solution treatment and precipitation hardening. The thermal conductivity is good and after hardening the electrical conductivity is about 80% I.A.C.S. One of the most important properties of the alloy is its ability to retain its strength up to temperatures of the order of 400° C.

There are certain limitations with regard to the size of material available as the alloy requires to be cast in relatively small moulds to achieve the best results. Care must also be exercised, in melting and casting, to avoid oxidation of the chromium, otherwise chromium oxide may be present which does not go into solution and may even prevent solution of the remaining chromium by forming films round it.

Heat Treatment

The normal solution treatment consists of heating to a temperature of 900°–1,000° C. for half an hour, followed by water quenching. With the higher temperatures somewhat better properties may be attained but there are practical difficulties to be considered. To avoid grain growth the shortest time at the lowest possible temperature is desirable although solution is more easily effected at the higher temperatures. With temperatures of the order of 1,000° C. there is a danger of parts sticking together in the furnace and, for preference, a temperature of 950°–975° C. should be selected.

The material is available in the solution-treated or solution-treated and cold-worked forms or it may be obtained fully treated. Rod material exhibits a variation in structure from delivery to delivery and even in the same delivery. Fig. 7 is a typical structure of ½ in. diameter rod in the " as-received " condition with a hardness of 128 D.P.N. Fig. 8 shows the structure of a similar rod with a hardness of 137 D.P.N. The remarkable difference in the grain size between the two samples is apparent. Fig. 9 is the same specimen as Fig. 7 after quenching from 930° C. when its hardness was 51 D.P.N. There is no appreciable change in grain size. Fig. 10 is the same specimen as Fig. 8 after a similar treatment. The hardness in this case was 65 D.P.N. and it will be noted that the massive grain of the material in the " as-received " condition has been broken up, although the grain size is still very large.

Precipitation hardening is carried out by heating to 475° C. for a period varying from 4–18 hours, depending on the amount of intermediate cold working. Figs. 11
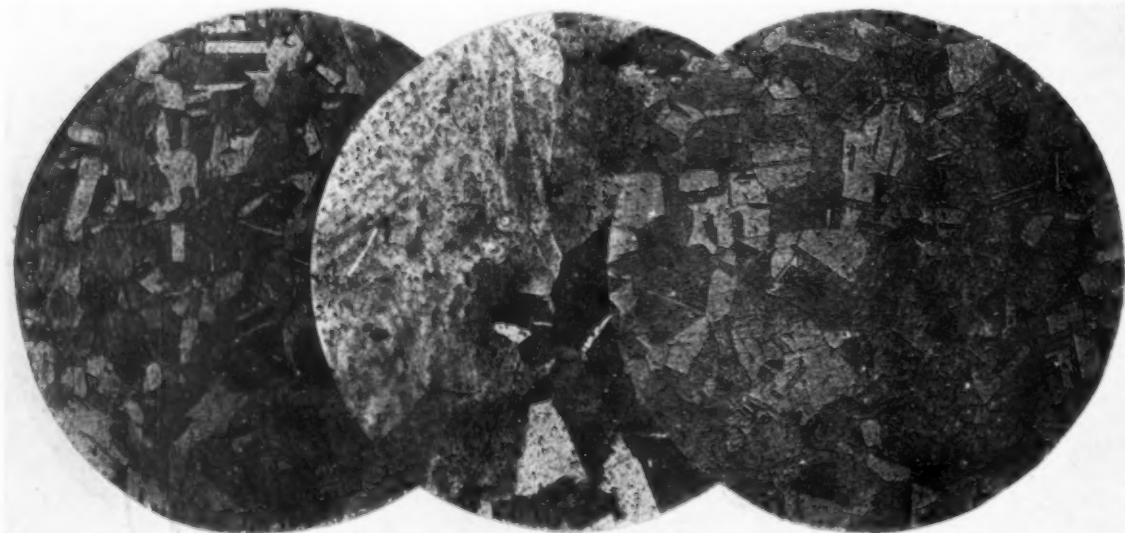

Fig. 7.—As received.   Fig. 8.—As received.   Fig. 9.—Quenched from 930° C.

Figs. 7, 8 and 9.—Structure of ½ in. diameter chromium copper rod after various treatments.   × 200

Fig. 10.—Quenched from 930° C.

Fig. 11.—Quenched from 930° C. and precipitation hardened at at 475° C.

Fig. 12.—Quenched from 930° C. and precipitation hardened at at 475° C.

Figs. 10, 11 and 12.—Structure of ½ in. diameter chromium copper rod after various treatments. × 200

and 12 show the structures of the specimens in Figs. 9 and 10, respectively, after precipitation hardening. The hardness values obtained were 120 and 125 D.P.N. Once again the large grain size is apparent in Fig. 12.

Somewhat higher values of hardness may be obtained by quenching from 1,000° C.

Properties

A typical composition for chromium copper is given below :—

Total chromium	..	..	..	0·4–0·75%
Chromium oxide	..	..	..	0·10% max.
Silicon ..	..	..	..	0·15%

Typical properties are listed in the Table below.

Condition	Ultimate Tensile Strength	Elongation % on 2 in.	Diamond Pyramid Hardness	Electrical Conductivity % I.A.C.S.
Solution treated	15	60	65	45
Solution treated and precipitation hardened..	29	25	142	82
As above but with 10% intermediate cold reduction.. ..	30	22	155	80
As above but with 25% intermediate cold reduction.. ..	35	14	160	80

Should the alloy become scaled in the course of heat treatment it can be dealt with in the same way as beryllium copper. It can also be electroplated quite readily.

OTHER HEAT-TREATABLE ALLOYS

There are also available several proprietary alloys which respond to heat treatment. Without attempting to deal with all of them, reference may be made to one or two examples. There is, for instance, an alloy of the following composition : Copper—72%, nickel—5·75%, aluminium—1·5%, zinc—remainder, which possesses a number of attractive features. This material may be

hot or cold worked, but for hot working there is a limiting temperature range of 800°–900° C. When quenched from 850° C. these alloys have a hardness of 60 D.P.N. and in this condition they may be cold worked. The material work hardens rather rapidly, however, and, after a moderate drawing operation, hardness values may rise to 140–210 D.P.N. Softened parts, heated to 500°–520° C. for 2 hours, reach a hardness of 180–210 D.P.N. The actual precipitation temperature should be varied to suit the degree of cold work to which the material has been subjected after solution treatment. As with other materials referred to, the question of solution-treatment temperature must be considered in relation to the possibility of excessive grain growth.

The copper-manganese-nickel alloys are also of interest as, by a combination of work hardening and heat treatment, mechanical properties second only to beryllium copper can be achieved and, as the ingredients are cheaper than beryllium they constitute a serious competitor for instrumentation. The electrical conductivity is also good, being about 80% of that for high conductivity copper. By suitable selection of composition and treatment, the hardness can be varied from 80–450 D.P.N., with corresponding tensile strengths of 24–85 tons/in.^{2}. In the hardest condition, the material is very brittle and can be snapped with ease.

This brief review of some of the copper-rich alloys which respond to heat treatment indicates the remarkable strides which have been made in the metallurgy of the copper alloys. It is reasonable to suppose that further progress will be made in the future to the benefit of industry in general.

At a recent meeting of the British Standards Institution, Technical Committee M.E.E./30—Ball and Roller Bearings, a decision was taken that the British Standard for tapered roller bearings should contain inch sizes only. British Timken, Ltd. have been asked to prepare a list of tapered roller bearings to comprise this range.

Alloy Steels for Plastic Moulds and Hobs

By L. Sanderson

Steels for forming plastic shapes under pressure must possess high surface hardness while retaining a tough core. Several have been developed, and are successfully used for this purpose, some of which are discussed.

NOTHING has been more spectacular than the discovery and development of plastics and their application to an ever-increasing number of purposes in the modern world. For this result to be achieved, however, it has been necessary for the steel manufacturer to carry out a great deal of research work so as to produce a range of steels capable of meeting the special requirements of this new and steadily expanding industry. It will be appreciated by those called upon to deal with plastics that the quality and finish of the moulded piece are largely governed by the way in which the mould impression is formed and finished. These impressions are made by one or other of two processes, hobbing or machining. Hobbing comprises pressing a master hob into the steel blank. When it is impracticable, machining must be employed.

Requirements

The moulds employed in plastics manufacture have to withstand severe pressures within the range 3,000–12,000 lb./sq. in., while in addition they must withstand elevated temperatures between 170° and 200° C. Some of the powders used in the making of these mouldings are highly abrasive, and this in itself necessitates certain special qualities of resistance in the mould steel.

Steels for plastic moulds must have a high degree of hardness on the surface, while at the same time possessing a core of " tough " character capable of resisting the shock and bending action experienced when plastic materials are moulded under pressure.

Some of the moulds are manipulated by hand and contain a number of loose parts. When the mould is assembled, and when the moulding is ejected, there is always a possibility of severe handling by the operator, resulting in damage should the mould be insufficiently hard. There is also a possibility of corrosion as a result of continual contact with organic powders of a corrosive type, among which may be mentioned urea- and thiourea-base moulding powders.

Many mouldings are required to have a smooth and lustrous surface, and this cannot be obtained unless the surface of the mould is itself highly polished, so that another requirement of the mould steel is the ability to take a high polish.

Summing up, a satisfactory steel for plastic moulds must have the properties outlined in the following notes. First, it must be machinable on a commercial basis, which means that, for preference, it should have a hardness represented by a Brinell hardness range of 150–250. Secondly, when made the mould should have a superficial Brinell hardness of 580–620, corresponding to a Rockwell hardness of C.58–60, and a V.P.N. of 700–750. This hardness should penetrate at least 0·002 in. below the surface, and should then fall off gradually towards the core, where the hardness should not exceed 500 V.P.N. (about 465 Brinell or

C.48 Rockwell). On the other hand, if an oil- or air-hardening steel is preferred, the surface hardness should be of the order of 465–495 Brinell (47–50 Rockwell C. or 500–550 V.P.N.).

The next requirement is that the central mass or core of the steel should have a tensile strength of 70–100 tons/sq. in. in addition to the highest possible impact resistance figure. The steel must also be of a type that does not readily oxidize or scale on the surface as a result of the hardening operation, not only because the mould has often to be made to extremely fine limits of accuracy, but also where the mould is of complicated design, polishing has to be carried out with a tool known as a *dolly*, and becomes expensive if a good deal of hard scale has to be removed. Moreover, the removal of this scale is liable to prevent the attainment of dimensional accuracy.

The heat treatment, and particularly the annealing treatment for machining, must be as easy as possible. It may be necessary at any time to alter the mould design slightly, which involves annealing the original mould so as to soften it sufficiently to allow the necessary machining to be carried out. Moreover, the steel must " move "—i.e., distort or change in volume—as little as possible as a result of the hardening process. This, again, is to ensure that the mould is as accurate as possible when hardened because, even if the moulded product is allowed certain tolerances, this allowance is usually required to accommodate any uneven shrinkage of the moulded part as it cools from the curing operation. There is thus no margin left to accommodate distortion or volume change resulting from hardening.

Typical Steels for Cut Moulds

A wide range of steels has been developed for plastic moulds and a number of typical compositions are given in Table I. Of these the first two are of the nickel-chromium-tungsten and nickel-chromium-molybdenum types, respectively, and may be used in the oil-hardened or case-hardened condition. The third steel is a stainless steel mainly used for cut moulds ; this again may be oil-hardened or case-hardened. The fourth composition refers to a high-carbon high-chromium steel of the type commonly used for non-shrinking steel dies designed for long runs.

The first two steels are the more generally employed for the reason that they present no difficulty in machining, despite the fact that even in the annealed condition they have a fair degree of hardness, while they conform closely to the requirements specified above for satisfactory plastic moulds.

The nickel-chromium-tungsten steel is widely used for cut moulds. Its hardness in the oil-hardened and tempered condition is about 480–495 Brinell (49–50 Rockwell C. or 520–540 V.P.N.), whilst in the case-hardened condition the figure is 700–760 V.P.N. In the

annealed condition, in which it is usually supplied, the Brinell hardness is 170–180. Electroplating of the dies is sometimes desirable when the urea moulding powders are employed in moulds of this material.

TABLE I.—COMPOSITION AND HEAT TREATMENT OF STEELS FOR MACHINED MOULDS

| Steel | Composition % | | | | | | Hardening Treatment | Tempering Treatment | V.P.N. at Surface |
	C	W	Cr	V	Mo	Ni			
1	0·3	0·5	1·0	—	—	1·3	O.Q. 820°–840°C. or Carburise in cyanide bath at 920°–940° C. O.Q. 820°–840°C.	200 °C. / 200° C.	520–540 / 700–760
2	0·25	—	0·8	—	0·5	2·5	O.Q. 820°–840°C. or Carburise in cyanide bath at 920°–940° C. O.Q. 820°–840°C.	200° C. / 200° C.	520–540 / 700–740
3	0·35	—	14	—	—	—	Carburises at 900°–950° C. O.Q. 950° C. or O.Q. 950° C.		700–750 / 520–530
4	1·5	—	12	—	0·75	—	Preheat to 750°–800° C. O.Q. 960° C. or Preheat to 750°–800° C. A.C. 1,020°–1,030° C.	200° C. / 500° C. / 200° C.	820–860 / 720–780 / 820–860

The nickel-chromium-molybdenum steel is used for the same type of moulds as the previous steel. The hardness values in the oil-hardened and case-hardened conditions are similar, whilst in the annealed condition the steel is slightly harder at 190–220 Brinell.

The steels are carburised at 920°–940° C. in a cyanide bath and then quenched in oil from 820°–840° C. Where the lower hardness value is sufficient, the carburising treatment may be omitted and the steel merely quenched in the oil bath.

The stainless mould steel, as mentioned above, is mainly used for cut moulds. It has a hardness of 520–530 V.P.N. in the oil-hardened condition and of 700–800 V.P.N. when case-hardened. It is supplied in the annealed condition with a Brinell hardness of 170–180.

The carburising treatment is carried out at 900°–950° C. in a cyanide bath and is followed by oil quenching from 950° C., which results in a hardness of 700–750 V.P.N. with a core hardness of 380–420 V.P.N. The hardness in the oil-quenched condition is 520–530 V.P.N.

The high-carbon high-chromium steel is used when the moulds are required to be hard throughout, and when the minimum distortion is essential. On the other hand, it is not suitable for moulds called upon to withstand shock or severe treatment in use ; injection moulds and sliding core pins are typical applications. It will not sink when used, but it has not a high impact value. It has the advantage that it will take a high polish, and it is also admirably wear-resistant. In the annealed condition, as supplied, it has a Brinell hardness of 230–250.

In heating to the hardening temperature, it should be preheated to 750°–800° C. Hardening may be effected by oil-quenching from 960° C. or by cooling in air from 1,020°–1,030° C. After tempering at 200° C. the hardness is 820–860 V.P.N., whilst a 500° C. tempering treatment results in a hardness of 720–780 V.P.N.

Hobbing Steels

The hobbing process as a means of producing moulds for plastic materials is mainly applied to those moulds with multiple cavities. Intricately formed impressions cannot be produced by hobbing, but ordinary plain outlines are readily reproducible with fidelity. So, too, are lettering and accurately spaced divisions on scales. Buttons and bottle caps are also produced by hobbing.

The steel used for the master hobs is forced into the mould steel at a pressure between 100 and 200 tons/sq. in. of projected design area and, in consequence, it must have a close grain structure, high strength and the ability to withstand wear. In addition, as the product is usually required to have a high polish, the hob must be able to take a high polish itself.

From the indication of pressures given above, it will be appreciated that master hobs have to withstand stresses of extreme severity and that moulds can only be made from steels which conform to the hobbing requirements. Typical materials are fully softened case-hardenable hobbing steels and fully softened nickel-chromium-tungsten steel of the composition given in Table I. The latter should, however, only be used for shallow impressions. There is also a fully softened stainless hobbing steel designed for use with the urea moulding powders, and commonly used for insert-button- and bottle-cap moulds.

TABLE II.—COMPOSITION OF STEELS USED FOR HOBBED MOULDS

Steel	C	W	Cr	Mn	Ni	Mo
1	0·1	—	—	0·4	—	—
2	0·1	—	14·0	—	—	—
3	0·1	—	0·6	0·2	1·25	—
4	0·15	—	1·5	0·45	3·5	—
5	0·15	—	—	—	1·5	0·3
6	0·3	0·5	1·0	—	1·3	—

In Table II a number of compositions of typical hobbing steels are shown. These are all hobbed in the fully softened condition when the hardness varies from about 100 Brinell, in the case of the unalloyed case-hardening steel, up to 170 Brinell for the nickel-chromium tungsten steel. Heat treatment consists of a carburising treatment followed by oil-quenching. The stainless steel may also be hardened by air-cooling from 1,020°–1,030° C. and, where the maximum hardness is not required, the nickel-chrome-tungsten steel may be merely oil-hardened, as mentioned in connection with cut moulds.

Hobs

For the master hobs themselves, users both in Great Britain and the United States favour a high-carbon high-chromium steel of the type already mentioned. Alternatively a carbon steel containing a percentage of tungsten may be used. A number of typical steels are shown in Table III.

Many steels have been put forward for master hobs, but those indicated represent the most widely used and accepted to-day. The high-chromium high-carbon- and the carbon-tungsten master hob steels are probably the most satisfactory because they can be hardened throughout, have an extremely dense structure, good resistance to compression and do not warp or " move " after being hardened.

General Remarks

It is not easy to include every possible requirement in regard to the heat treatment of these various steels, but an attempt has been made to outline as simply and

Steel	Composition %						Hardening Treatment	Tempering Treatment	V.P.N. at Surface
	C	Mn	W	Cr	Mo	Ni			
1	1·5	—	—	12	0·75	—	Preheat to 750°-800° C. O.Q. 960° C. or Preheat to 750°-800° C. A.C. 1,020°-1,030° C.	200° C. 500° C. 200° C.	820-860 720-780 820-860
2	0·5	0·4	—	0·8	0·5	1·5	O.Q. 930°-960° C. or Carburise in cyanide at 900°-950° C. O.Q. 950° C.	—	520-530 700-750 (case). 380-420 (core).
3	0·9	1·2	0·5	0·5	—	—	Carburise or cyanide at 930° C. Quench in water or brine at 800°-900° C.	160°-260° C.	C. 63-57 Rockwell
4	1·2-1·3	0·2-0·3	4-5	0·7-0·8	—	—	Carburise in cyanide at 930° C. O.Q. 760°-780° C.	—	C. 62-57 Rockwell

briefly as possible the most generally satisfactory procedure.

A few general remarks on these steels may prove helpful to users. The steel must be as free as possible from interior flaws liable to be exposed when the impressions are cut, and so to spoil, if the mould or die is used, the finished appearance of the moulding. Even when these flaws are so small as to be invisible to the naked eye, or negligible in extent, they have a habit of enlarging under service conditions and so forming defects or furrows on the surface of the moulding. The unalloyed steels are, of course, much softer, when annealed, than alloy steels containing the same percentage of carbon. It will be appreciated, therefore, that although a steel of unalloyed type is readily hobbed, a mould produced from it will have a lower strength than a harder steel. Too hard a mould steel means that the hob encounters too great a resistance when pressed into the mould and, in consequence, produces too shallow an impression. Too soft a steel means rapid wear of the moulds and inadequate sharpness of the impressions, besides a danger of collapse. The steel chosen must, therefore, represent the happy mean between these extremes.

Machine-cut moulds call for a steel that presents few, if any, machining difficulties. While the correct annealing treatment may make this possible, the temptation to make machining easier by using free-machining qualities of steel should be resisted.

The primary requirements are strength and resistance to wear. Wherever the mould has thin sections or has a considerable mass, it must be strong, so as to prevent the high temperature and pressure from completely driving in the surface. If a case-carburising steel is used for massive plastics moulds, it must be of a type capable of developing a high compressive strength at the core. The factor of high resistance to wear becomes increasingly important in proportion as the run is longer and, where this factor is important, it is preferable to employ steels of alloy type.

The heat-treatments of these steels have been very barely outlined, and a little amplification is called for. Quite apart from the ordinary precautions to be observed particular care must be taken to minimise warping and volume change, and to eliminate as far as possible all oxidation, scaling and decarburisation of the surface of the tools.

If it is desired to forge any of these steels, care should be taken that they are brought slowly to the forging temperature, which is usually within the temperature range of 930°-1,100° C. The maker should be consulted as to the precise forging temperature for any particular steel, as much will depend on composition, size and amount of reduction at any one blow. Forging should not be carried on below 870° C. with most of these steels, although the less highly alloyed steels can continue to be forged at as low as 820° C., and the unalloyed steels down to 760° C. ; here again, the manufacturer should be consulted. Most of the steels (the high-carbon, high-chromium steels are exceptions) will be better for a normalising treatment after forging, and slow heating to the requisite temperature is essential. This temperature lies within the range 840°-900° C. The temperature should be maintained for a period ranging from ¼-1 hour according to mass. For annealing, slow and even heating to a temperature within the range 760°-900° C., and maintenance at this temperature for from 1-4 hours, is required. Slow cooling is required in all cases, and for the higher alloyed steels should on no account exceed 25° C. per hour down to 540° C. For both these heat-treatment processes, exact temperatures for particular steels should be obtained from the steel manufacturers.

Hot Work Tool Steel

A NEW tool steel, especially developed for hot work, has been introduced by Allegheny Ludlum Steel Corp. which is claimed to have excellent resistance to shock and abrasion at elevated temperatures. The good hot hardness characteristics of the alloy make it suitable for many hot work jobs, but it is specifically recommended for applications that require good toughness at relatively high hardness or where abrasion resistance and resistance to heat checking are important. Such applications include brass extrusion dummy blocks and dies, valve extrusion die inserts, forging die inserts, forging press dies and hot punch tools.

Approximate chemical analysis of this new steel is as follows : 0·40 carbon, 0·35 manganese, 0·25 silicon, 4·25 chromium, 4·25 tungsten, 4·25 cobalt, 2·25 vanadium and 0·40 molybdenum.

Results of Charpy V notch impact tests at elevated temperatures show values of 7¾ ft.-lb. at 204° C., 6½ ft.-lb. at 316° C., 12 ft.-lb. at 427° C., 9 ft.-lb. at 538° C. and 8¾ ft.-lb. at 649° C.

Exactor, Ltd.

FOR the past 16 years " Exactor " has been associated with a range of specialised hydraulic control and other equipment. The Exactor Control Co. has acquired the exclusive manufacturing and sales licence for the well-known American " Trabon " system of centralised lubrication.

The hydraulic experience of this company, allied to the fund of specialised knowledge of lubrication enjoyed by their associated company, Messrs. Dualloys, Ltd. bearings, make this a particularly logical and satisfactory extension of their activities. In keeping with this development the Exactor Control Co., Ltd., has accordingly changed its name to " Exactor. Ltd." The address and telephone number remain the same : 108, Park Street, London, W.1, Tel : MAYfair 0252.

The Annealing of Nickel and High-nickel Alloys

In the first part of this article, published in our February issue, various aspects of the annealing of nickel and high-nickel alloys were discussed, including soft annealing, dead-soft annealing, temper annealing and stress equalising annealing. This concluding part deals with bright annealing, age-hardening of " K " Monel and various auxiliary factors in annealing.

BRIGHT ANNEALING

For true bright annealing it is now generally accepted that the atmosphere must contain definite proportions of the reducing constituents carbon monoxide or hydrogen, as well as being free from oxygen. Reducing atmospheres of this kind are mostly derived from anhydrous ammonia or fuel gases, principally coal gas.

For pure nickel, a mildly reducing atmosphere is sufficient, but for Monel a somewhat more reducing atmosphere is required. Partially burnt coal gas containing about 8% each of hydrogen, carbon monoxide and carbon dioxide is satisfactory.

With Inconel there may be some difficulty in obtaining the same white surface which results from bright annealing Monel and nickel. This is because the high affinity of chromium for oxygen causes the formation of chromic oxide in the presence of carbon monoxide, carbon dioxide or water vapour. Consequently, a strong, closely adherent oxide skin is formed : this is, in fact, one of the inherent advantages of Inconel and has led to its wide use at high temperatures.

For Inconel, the recommended atmosphere for the best effects is cracked ammonia which is of adequate purity providing that the required dryness is maintained by the use of metallic muffles. Though the cost of the protective atmospheres, together with that of muffle maintenance, may be high, the process is often economically justified.

AGE-HARDENING OF " K " MONEL

" K " Monel is an age-hardenable alloy which responds to thermal hardening of the precipitation type, the extent of improvement depending upon the condition of the material, the hardening temperature, soaking and rate of cooling. By control of these factors hardness can be varied from 160–380 D.P.N.

Six standard grades are available as summarised below :—

Grade A.—This is in the hot-rolled, extruded or forged condition, whether finally annealed or not. If reasonably constant properties are required on hardening, Grade A " K " Monel should be finally annealed.

Grade B.—This is Grade A material which has been hardened by thermal treatment.

Grade C.—Is material in the cold-worked condition, not thermally-hardened.

Grade D.—Is thermally-hardened Grade C and is the hardest form in which " K " Monel is available.

Grade E.—Is material which has been cold-worked and annealed.

Grade F.—Is thermally-hardened Grade E.

Table VII summarises temperatures and times required for annealing, age-hardening and stress-equalising.

The metal must remain at the appropriate temperature for the correct period and is then quenched in water, or

TABLE VII.—HEAT TREATMENT OF " K " MONEL FOR MAXIMUM PROPERTIES

Treatment	Material, Condition or Grade	Metal Temp. ° C.	Time at Temp.	Rate of Cooling
Regular anneal for softening	Cold-worked.	870	2–5 min.	Quenched whenever possible. Sections over ¼ in. must be quenched.
		980	1–3 min.	
Age-hardening treatment.	Grade A or Grade E soft (150 – 190 D.P.N.), hot-rolled or cold-worked and annealed.	580–590	16 hr.	Average 8°–14° C. per hour down to 482° C. *Note.*—Cooling down to 482° C. may be in steps of 55° C. holding furnace 4–6 hours at each step. Cooling below 482° C. to room temperature may be done without regard to the cooling rate.
	Grade C medium hard (190 – 260 D.P.N.), cold-worked.	580–590	8–16 hr.	
	Grade C full hard (260 – 340 D.P.N.), cold-worked.	530–540	6–10 hr.	
Stress-equalising treatment.	Material cold-worked after age-hardening (Grade D) or cold-worked parts which are *not* to be age-hardened. (Grade C).	300–345	1–2 hr.	Quenched or furnace-cooled.

preferably in a solution of 2% by volume of denatured alcohol in water. Partial softening can be carried out, if required, on cold-worked or thermally-hardened material by heating to 590°–790° C., depending on the degree of softening required.

Full hardening of " K " Monel is effected by a single heat treatment, consisting of ageing at the required temperature. Maximum response is obtained if ageing is followed by controlled, slow cooling, but sufficiently good response can be obtained by allowing the material to cool down in the furnace. The material should not be raised above the recommended temperature, otherwise there will be some loss of final hardness.

AUXILIARY FACTORS IN ANNEALING
Pyrometry and Temperature Control

Accurate control of annealing temperature and of its uniformity are fundamental if the best results are to be obtained. Satisfactory indicating-controller and controller-recording pyrometers are available and their use ensures good results. Recommended noble and base-metal thermocouples and the necessary protection tubes are summarised in Table VIII. Iron-Ferry and Chromel-Alumel thermocouples should be changed at least every three months. Noble metal thermocouples may be used as long as their accuracy is not impaired. The accuracy of all thermocouples should be checked daily.

In both open- and closed-annealing, the thermocouple should be located as near the work as possible. In box heating care must be taken to see that the thermocouple

Thermocouple	Maximum Temperature		Protection Tube	
	For Prolonged Operation		Reducing Atmosphere CO or H_2	Oxidising Atmosphere O_2[1]
	° C.	° F.		
Platinum vs. Platinum (90%)—. . } Rhodium (10%) . . }	1370	2500	(Porcelain (Mullite[a]))	None
Chromel vs. Alumel . .	1090	2000	Mullite	None
Iron vs. T.C. Ferry . .	870	1600	Nickel or Inconel	None

1 Although bare wire may be used, a protection tube decreases oxidation, especially with the Iron/Ferry thermocouples and to a lesser extent with Chromel/Alumel thermocouples.

2 The use of a porcelain primary tube inside the Mullite tube is recommended in reducing atmospheres at temperatures above 870° C. Nickel seamless tubing with one end closed by welding is generally used to protect the tubes from mechanical shock. The thermal conductivity of nickel is sufficiently high to prevent undesirable "lag."

is located inside the box and at about the centre of the charge. The temperature should be regulated by means of the thermocouple in the furnace chamber with the second thermocouple in the charge connected to a recorder so that the difference in temperature between the furnace and the box can be noted.

Control is not difficult in electric furnaces, but additional provision may be required for the circulation of gases in gas-fired furnaces. Whether they are heated by gas or electricity, furnaces so fitted give more uniform results. If grain size is to be controlled the furnace temperature should at no time exceed the recommended annealing temperature.

In general, low temperature treatment (up to 700° C.) generally can be carried out with greater accuracy and economy in forced-air circulation furnaces than in any other type.

Protective Atmospheres

The function of a protective atmosphere, as the term implies, is to prevent or at least to restrain within tolerable limits, deterioration of the surface of the metal being treated. The required degree of control depends upon the results desired. While bright annealing, as already described, can be done only in the absence of all oxidising influences, many parts may be annealed by a process which will develop a superficial tarnish or oxide coating. Such parts include those that are to be polished after annealing, those that do not require a perfectly clean surface, or those that are to undergo further fabricating processes. It should be noted that highly stressed parts such as springs and diaphragms should be kept free from heavy oxides, although tarnishing and superficial oxidation do not impair their performance.

Whatever the final use of the parts, high-nickel materials should never be annealed in a strongly oxidising atmosphere, as the heavy oxide coating so produced, even though it be removed by grinding or pickling, may be harmful to the underlying metal.

While prevention of scaling is the primary object of using a protective atmosphere it is often of equal importance to ensure that avoidance of oxidation does not of itself set up objectionable secondary effects. Principal requirements of the protective atmosphere are : (1) An overall composition such as to be non-oxidising to the material under treatment, and (2) adequate freedom from sulphur and other undesirable impurities.

The most commonly used protective atmospheres are derived from anhydrous ammonia or fuel gases, principally coal gas.

Cracked Ammonia.—From ammonia, atmospheres may be obtained by cracking into its constituent elements, the product containing three parts of hydrogen with one of nitrogen. Alternatively, the cracking may be followed by partial combustion, the product after condensation of water vapour then consisting of nitrogen with between 2 and 15% of hydrogen, according to the ratio of cracked ammonia : air employed for combustion. Further drying is accomplished by passing the gas through activated alumina or silica gel dryers.

These atmospheres are of exceptional purity, being quite free from sulphur and other deleterious constituents. In the case of the cracked gas, the water vapour content is extremely low and the resulting high hydrogen : water-vapour ratio makes it ideal for bright annealing of Inconel. The cost of ammonia is, however, fairly high and use of the cracked or burnt gas is not as a rule justified except for small-scale operation. In the case of the burnt gas this difficulty may be mitigated by the use of a regenerative system.

Coal or Coke-oven Gas.—Atmospheres derived from coal or coke-oven gas by partial combustion followed by removal of excess water vapour are very widely used. They are cheap and as coal gas is readily available, convenient in use ; moreover, the composition may be adjusted by
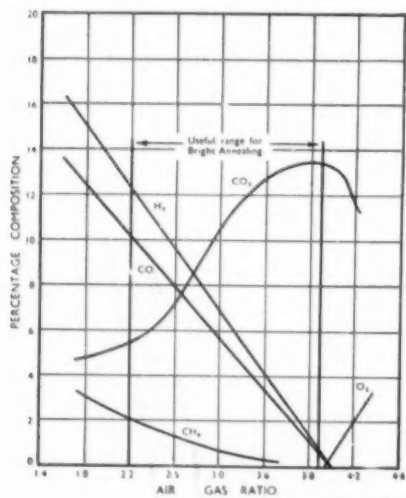

Fig. 15.—Flame method for purging a heat-treating box.

Feel Top to determine when flame has gone out

Gas burning freely under Box Top

Escaping Products Combustion

Gas Inlet

Fig. 14.—Composition of partially burnt coal gas.

Courtesy of Birlec Ltd.

Hose Connection

Outlet Tube from Furnace or Box

U Tube Manometer

Light Escaping Gas

Orifice or Jet

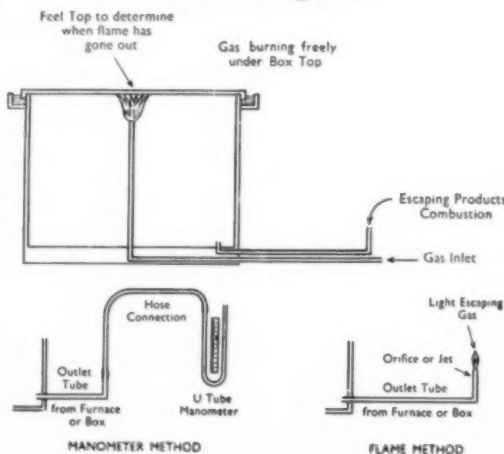Outlet Tube from Furnace or Box

MANOMETER METHOD

FLAME METHOD

Fig. 16.—Methods for controlling gas pressure in the furnace or annealing box.

variation of the combustion ratio. Fig. 14 shows the range of product analysis available from a typical coal gas of 450 B.Th.U./cu. ft. calorific value.

Similar mixtures are obtained by partial combustion of hydrocarbon gases such as propane or butane, or of liquid hydrocarbon mixtures such as paraffin, and these may be used where coal gas is not available.

Purification.—For atmospheres derived from fuel gases some measure of sulphur purification is necessary. This is accomplished by the removal of hydrogen sulphide in an iron oxide purifier box, followed by removal of residual organic sulphur (carbonyl sulphide) from the product gas. Alternatively, the raw coal gas may be submitted to a catalytic purifying treatment, which removes most of the sulphur before feeding to the burner unit. One such process employs a nickel sub-sulphide catalyst at a temperature of 200°–300° C., which converts the organic sulphur in the coal gas (except that present as thiophene) to sulphur dioxide and hydrogen sulphide which are then removed by alkaline scrubbing and iron oxide treatment, respectively.

Purging.—Having a source of clean, dry, oxygen-free hydrogen or cracked ammonia, and having eliminated formation of water vapour within the equipment, it is necessary to ensure thorough purging of air and other undesirable gases from the furnace chamber or heating box.

When using cracked ammonia or hydrogen in batch-type furnaces, pre-purging with an inert gas such as completely combusted town's gas or cylinder nitrogen is essential. If this precaution is omitted, sooner or later an explosion will occur.

Boxes can be purged by the following procedure, which is illustrated in Fig. 15.

1. Place the charge in the box, allowing ample space for the circulation of gas between the parts. See that the thermocouple is properly located.

2. Before placing the cover on the box, admit hydrogen to the box through the inlet tube and light the flowing gas. Adjust the flame to a length of about 1½ in. The flame will then be burning against the top.

3. Place the cover on the box and seal it thoroughly with sand or clay. The outlet tube at the bottom of the box should be left open to permit the escape of air and the products of combustion.

4. When air has been removed completely the hydrogen flame will be extinguished. This becomes apparent by cooling of the box which can be detected by feeling with the hand at a point just above the flame.

5. Connect a manometer to the outlet without interrupting the flow of gas and regulate the flow so that the manometer will show a positive pressure within the box of not less than 0·2 in. of water, at which there will be a slow escape of gas bubbles through the water in the tube. Then place the box in the furnace for the annealing operation.

An alternative method of regulating the pressure is to light the gas escaping from the outlet tube, instead of connecting it to a manometer. The flame should be 1–2 in. long. In order to accomplish this without excessive gas consumption the end of the line should be fitted with an orifice, jet or valve. This method, illustrated in Fig. 16, is, however, less reliable and less economical than the manometer method.

After annealing, the box may be cooled in the furnace or may be withdrawn and cooled more rapidly in air. Care should be taken to increase the flow of reducing gas to maintain the gas pressure at 0·2 in. or 1–2 in. length of flame. When the box has cooled below 260° C. there is no danger of oxidation and the work may be removed.

Effect of Cold-work Previous to Annealing.—The effect of cold-work is important and must not be overlooked if the best results, especially control of grain size, are to be obtained. The greater the amount of cold-work to which the metal or alloy has been subjected, the lower is the annealing temperature required to produce the same degree of softness without increasing grain size, or the shorter the required time at temperature. Hard Monel that has been cold-worked heavily
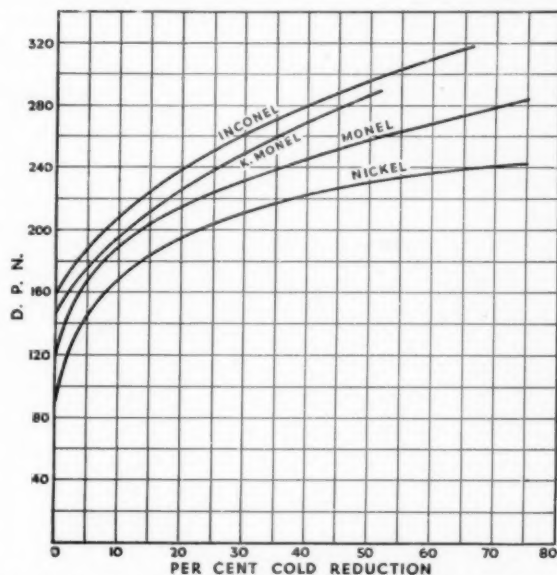

Fig. 17.—Increase in hardness of nickel and high-nickel alloys with cold-working.

(50% or more) softens more rapidly than half-hard Monel that has been cold-worked approximately 25%.

The amount of previous cold-work also has a critical influence upon the ductility of nickel and nickel alloys after annealing. This is true regardless of the type of cold-work. If only a small amount, say 10%, is done, the full ductility cannot be restored by annealing, even though the hardness is reduced to the level of soft material. A minimum of approximately 20% of cold-working between anneals is required to ensure adequate ductility and softness following annealing.

Internal fracture is liable to occur if a metal or alloy is excessively cold-worked. This cannot be eliminated by annealing and will cause the work to be scrapped. Monel, nickel and Inconel sheet and strip should not be cold-worked more than 50% reduction in thickness by spinning and deep-drawing operations.

The curves in Fig. 17 show rates of hardening due to cold-working for these materials and it will be seen that they harden more rapidly than softer materials such as aluminium and copper. Because of this, the fabricator will seldom be able to do more than 50% cold-work between annealing operations, since the metal will

become too hard for easy forming. The approximate hardness relationships for nickel, Monel and Inconel are given in Fig. 18.

Effect of Rate of Cooling

Neither slow cooling, whether in or out of the furnace, nor rapid cooling by quenching, have any specific effect on the softness of annealed nickel, Monel and Inconel. Therefore, rapid cooling is preferable both to save time and to minimise oxidation.

" K " Monel differs from nickel, Monel and Inconel as it ages or precipitation-hardens during slow cooling. It must, therefore, be cooled rapidly by quenching from its annealing temperature.

Grain-size Control and its Effect upon Fabrication

Coarse-grained material is unsuitable for most cold-working operations, especially those involving large amounts of cold-work. Grain refinement by methods akin to the normalising of steel is not possible with nickel, Monel and Inconel. A coarse grain in the high-nickel materials cannot be refined by thermal treatment only ; it can only be removed by cold-working in excess of 50%, followed by recrystallisation as a result of soft annealing.
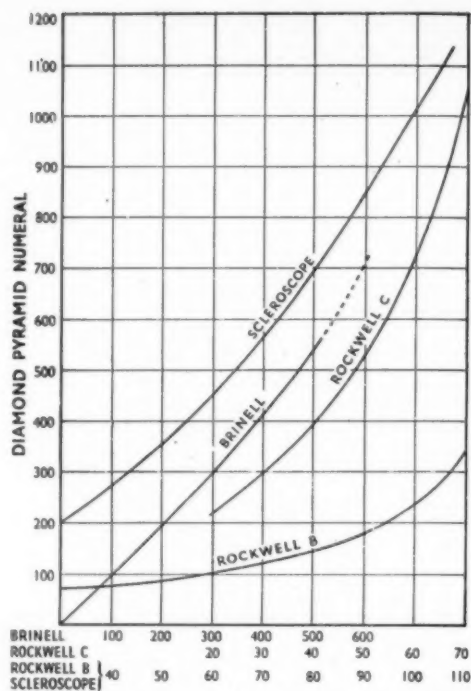
Fig. 18.—Approximate hardness relationships for nickel and high-nickel alloys.
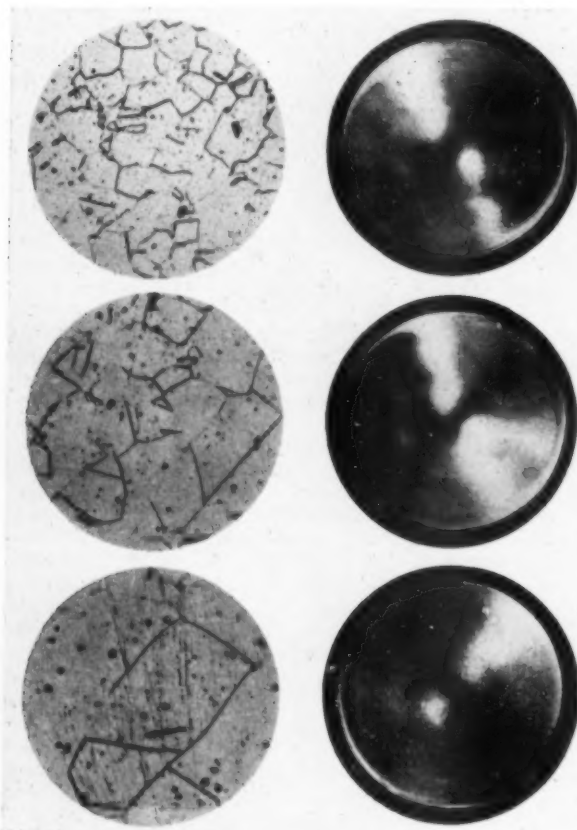
Fig. 19   Fig. 20

Fig. 19.—Photomicrographs (100 ×) and photographs (three-quarter size) of Monel sheet.
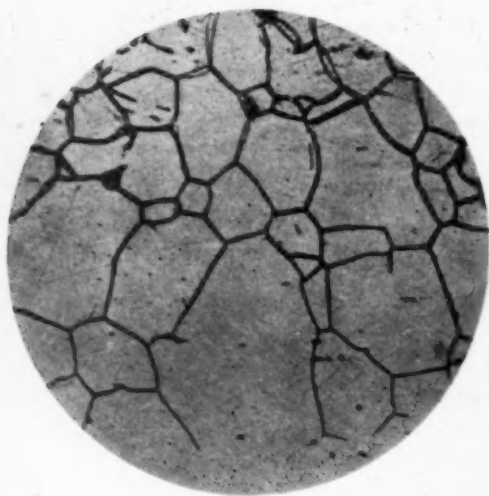
Fig. 20.—Photomicrograph (100 ×) of nickel sheet showing intercrystalline attack resulting from heating in an atmosphere which fluctuated alternately from oxidising to reducing.

Maximum workability is obtained with material that has been annealed to remove completely the effect of previous cold-working, without allowing appreciable grain growth to occur. This condition is described as " soft temper, fine to medium grain." The actual grain size should not exceed 0·0025 in. (0·064 mm.). This gives the best combination of ductility to permit extensive deformation, strength to withstand the action of the tools, and surface quality to facilitate polishing. Only in those cases in which the final surface condition is unimportant may grain size and its control be neglected.

The photomicrographs and photographs in Fig. 19 illustrate fine, medium and coarse-grain sizes in Monel sheet and the surfaces of Erichsen ductility test cups produced from them. Similar results are obtained with nickel and Inconel sheet and strip.

The Effect of Fluctuating Atmospheres during Annealing

If nickel and the high-nickel alloys are heated in atmospheres that fluctuate between oxidising (excess of air) and reducing (excess of carbonmonoxide or hydrogen), severe intercrystalline attack with resulting embrittlement will occur even though the atmosphere is free from sulphur. This is shown in Fig. 20. This type of embrittlement can be prevented by maintaining a constant and sufficient excess of a reducing atmosphere during heating and cooling.

Protection from Contamination

Many lubricants used in deep-drawing and spinning operations contain sulphur and lead, which must be removed before annealing to prevent the occurrence of embrittlement. Whatever the type of lubricant, it should be thoroughly removed from the material by washing with trisodium phosphate or with volatile, organic, grease-removing solvents such as carbon tetrachloride or trichlorethylene. Paint marks and other adherent substances should also be removed.

Although the main sources of discoloration of annealed nickel and nickel alloys are either incorrect furnace atmospheres or oil and dirt on the material, there are other possible sources that should be considered, as, for example, spallings from furnaces and boxes and " pick-up " from contact with improperly cleaned surfaces.

Parts that are to be heated directly in a furnace should not be placed on the hearth. Either the hearth should be covered by a sheet of nickel or of a heat-resisting alloy, or the parts should be placed in a tray or basket for handling in and out of the furnace. Protection should also be provided to prevent damage by particles falling from the furnace roof, either by means of a cover or by installing a metal roof lining.

Inconel and other heat-resisting nickel-chromium-iron alloys are particularly suitable for furnace linings, trays, baskets or sealed boxes.

Acknowledgment

Costing and Modern Accounting Methods in the Metal Industries

IV.—Recording and Control of Capital Expenditure

By S. Howard Withey, F.Comm.A.

It is important that the method of determining the value of stock should not distort the real trend of trading results. In this article the author deals with this point at length in addition to considering various aspects of capital expenditure, including depreciation.

STOCKS of materials and stores under a particular heading can usually be verified and compared with the book records at least once every three months, and if the stores ledger is kept up to date the daily routine will not be disturbed even though a number of adjustments may have to be made. For some time, the Inland Revenue authorities have been insisting that, for income-tax purposes, metal stocks should be assessed at the lower aggregate of cost or market value—i.e., that the *lower total* on the basis of either cost price or market price should be shown on the credit side of the periodical trading or profit and loss account and on the assets side of the balance sheet. Consequently, as the question of stock value for purpose of taxation is always one of fact by decision of the Commissioners, some metal manufacturers and traders who adopted the " pick and choose " method, and valued each item of stock on it own merit have been compelled to adjust their inventories. The Council of the Institute of Chartered Accountants has recommended that a consistent basis should be adopted and that normally this should be the lower of cost or market value. If direct or overhead expenditure is included as part of the cost of finished or partly processed products, such expenditure should be restricted to the amount which had been incurred in bringing the stock to its existing condition and location, thereby showing a fair view of the trend of results.

Calculations of market value are usually based on prices which it is estimated would be realised after allowing for any expenditure that would have to be incurred before disposal, with due regard to abnormal and obsolete stocks and the prospects of disposal. When stock values are arrived at by reference to the cost of replacement, in no case should the market value be exceeded, and it should be stated on the balance sheet that the stock has been valued " at the lower of cost or replacement value."

Tin and rubber producing companies usually value stocks of products at the prices subsequently realised *less* the selling costs, and this fact is nearly always

	£		£
To Opening Stock	15,000	By Sales, net	100,000
,, Trade Purchases	50,000	,, Closing Stock	15,000
,, Productive Wages	30,000		
,, Gross Profit	20,000		
	£115,000		£115,000

indicated in the final accounts, and when stocks of by-products—the actual cost of which cannot be determined—are valued at the prevailing sale prices, after deducting all expenses incurred before disposal, the cost of the main product may be reduced accordingly. Whatever basis is adopted for ascertaining cost or calculating market value, it should not distort the view of the real trend of trading results.

The recent case of *Commissioners of Inland Revenue v. Cock, Russell & Co., Ltd.* cannot fail to induce many traders to deflate their inventories by adopting the " pick and choose " method, particularly if by so doing they can, during a period of falling prices, show a considerably smaller figure in their final accounts under the heading of Stock-in-trade. This case decided that any basis which is considered prudent by normal businessmen may be taken for stock valuation purposes, and while it is true that there are no rules in the provisions of the income-tax act as to how trading stocks should be assessed, there is always the need for maintaining sound accounting and commercial principles for the determination of gross profits which is, of course, one of the main objects of pricing inventories.

It has been suggested by the Council of Retail Distributors that the Inland Revenue should permit each item of stock to be valued by the taxpayer on its own merits, either at cost price or market price, whichever the trader may choose. The method of dealing with trading stocks in the income-tax legislation of the Commonwealth of Australia will no doubt receive the consideration of the Tucker Committee, which is enquiring into the method of computing trading profits for the purpose of charging them to income tax, and is expected to report upon any alterations of the tax law which may be desirable. When considering whether any special rules are required for the valuation of trading stocks the possible effects on trading results should not be overlooked.

In this connection, the desirability of pricing out on the basis of cost was emphasised in a case in which a Lancashire firm of manufacturers made a gross profit of 20% on return during the twelve months ended December last. This firm had a stock of partly-finished products which had cost £15,000 and the partners decided to use material of a different grade and to produce a better quality product. The invoiced cost of the trade purchases for the year was shown in the books at £50,000 and during the same period the productive wages amounted to £30,000. After allowing for returns inwards and allowances made to customers in lieu of actual returns, the sales for the year totalled £100,000, and as all the new material had been used the audited trading account disclosed a gross profit of £20,000, as is shown above.

At the close of the previous year, the stock on hand had been entered on suitably ruled sheets, one assistant being detailed to call out the quantities and descriptions of the partly-finished products lying in the workshops, and these particulars, together with an indication of the stage of manufacture reached, in each instance, being set down by another person. All calculations and extensions had been carefully checked by reference to invoices, time sheets and progress reports, and the actual pricing out of the various items had been completed by the manager of the buying department, whose certificate appeared in the stock book directly above the firm's certificate.

As the stock had not been worked on during 1949, the current value was ascertained by pricing each item at both cost price and prevailing market value, the latter amounting to only £12,000 (in round figures). If this figure had been credited to the Trading Account, the balance of gross profit shown in the account would have been £17,000, and this would obviously have been incorrect as the actual gross profit was £20,000—viz., sales, £100,000 *less* cost of manufacture, £80,000.

The general conclusion to be drawn, therefore, is that in the interests of accurate accounting, gross profits should be arrived at after assessing the value of trading stocks at cost price, and that any reduction in stock values should be provided for and shown as a reserve when computing the balance of taxable profit.

The process of stocktaking is somewhat tedious, but much depends upon the accuracy of this work. When the details are set down and classified to show separate totals for incorporation in subsidiary trading accounts, particular care is called for, to ensure that no items of the following nature are included :—

(*a*) Assets such as thermostats, pyrometers, energy regulators, analytical balances, etc., the cost value of which is shown in separate accounts in the private ledger. To include such assets on the stock sheets would cause an inflation of the credit side of the periodical profit and loss account and also of the assets side of the balance sheet.

(*b*) Materials such as manganese briquettes, cements, refractories, etc., which, although received and taken into stock have not yet been debited to purchases account. Unless the invoice or other debiting document has been recorded in the books, the material should not be regarded as forming part of the stock.

(*c*) Metal products which have been sold and passed through the sales book but which, for any reason, have not yet left the premises. No products can be treated as sales and stock without causing an inflation of the credit side of the trading account.

As a rule, the major part of the trade purchases is transacted on a credit basis, involving the use of an analytical type of purchases journal. The net invoiced cost prices are credited to the respective personal accounts, and the monthly or other periodical totals debited to specific accounts, and unless a proper distinction is drawn between expenditure to be charged against

ELECTRIC PLANT

Description : ....

Location : ....

Fitments : ....

Maker's Name : .... No. : ....

Travelling Wheels : ....

Clutch : .... diam. .... wide.

Motor : .... B.h.p. .... volts .... amps .... phase.

Estimated Service Life : .... years.

Rate of Depreciation : .... per annum.

Cost, viz. :—	£	s.	d.	Remarks
Acquisition				
Carriage				
Accessories				
Installation				
Total Cost				

Back of Card

	£	s.	d.	REPAIRS AND RENEWALS		
Value as at 19				Date	Particulars	Cost
Less Depreciation written off						£ s. d.
Diminished Value						
Additions or Improvements						
Value as at 19						
Less Depreciation written off						
Diminished Value						
Additions or Improvements						
Value as at 19						
Less Depreciation written off						
Diminished Value						
Additions or Improvements						
Value as at 19						
Less Depreciation written off						
Diminished Value						
Additions or Improvements						

profits and the outlay which has had the effect of increasing the number or total value of the trading assets, the final figures are bound to be inaccurate and misleading.

When a metal business is acquired as a going concern, with the business premises, furnaces, instruments, lighting equipment, tool room, machinery, plant, accessories, fixtures and fittings, components and stocks, goodwill, etc., the entire expenditure should be treated in the accounts as capital expenditure and should be classified under definite headings for separate treatment. The circumstances surrounding each item must be taken into consideration, and the system of accounting should be framed to facilitate the debiting of cost prices to the proper accounts.

The cost of acquiring freehold or leasehold works premises is, of course, a capital charge, and if any legal expenses are incurred by the purchaser in connection with the transfer of the property, such expenses may be debited to the asset account and included in the amount shown on the balance sheet. It is advisable, however, to write off legal expenses as soon as possible, this being effected by crediting the property account and debiting a legal and professional charges account, the final balance of the latter being charged against the periodical profits ; any payments which add to the capital cost of a profit-earning asset or section of equipment may be capitalised for the time being.

While repairs to works or mill premises constitute a direct charge against the revenue, if the repairs increase the capital value as between a willing buyer and a willing seller the cost involved should be treated in the accounts as capital expenditure. The full invoiced cost prices of new furnaces or additional plant and machinery should be debited direct to a separate asset account opened in the private ledger, as also should the carriage

or transportation charges incurred by the purchaser up to the time of the arrival of the asset. Installation or foundation expenses may also be capitalised and, in those instances where it is found necessary to undertake repairs or adjustments to ensure immediate efficiency, the total cost of such repairs, etc., may be debited to the private ledger account.

When a machine or section of plant is replaced by equipment which has been designed to guarantee greater productive efficiency at a lower cost of operation, the book value of the old asset should be written off and the capital cost of the new equipment debited to the asset account. It is the practice of some metal traders to allow the book value of old and original assets to remain open, and when this is the case the full cost of replacements may be treated as a charge against profits. The cost of installing telephones and electric lights, etc., is a capital charge, but all expenses incurred in maintenance should be shown on the debit side of the periodical profit and loss account and included in costs.

Mechanisation in all sections of the metal industry demands the constant presentation of information disclosing the efficiency of the various types of machinery and plant, and while in theory each productive installation should operate at 100% efficiency, this is rarely achieved in practice. During recent years, manufacturing processes have been revolutionised and output speeded up, and in many departments whole layouts have been greatly simplified as the result of many years of research and experience.

Nevertheless, the ultimate wearing life of a piece of machinery is a problem calling for special knowledge and an appreciation of a variety of factors, and fortunately, under the supervision of thoroughly qualified technicians, many suppliers have been able to develop first-class organisations for the purpose of keeping maintenance costs within reasonable limits. Unless, however, a systematic control is exercised over different units and groups of equipment, it is extremely difficult to determine the correct figure of depreciation to be included in costs and contracts, and equally difficult to form proper decisions regarding the installation of special or additional equipment to meet current requirements.

It is highly desirable that records of machinery, plant and tools should provide the amount and kind of information required by engineers and executives, and in order to be of real practical utility all equipment of the same type, or which is likely to be used for similar purposes, should be grouped together and the figures summarised to show at a glance the precise number of units of each class and all additions, improvements and any other variations which have taken place during the particular accounting period under review. When kept properly and up to date, machine and tool records enable economies to be effected ; for instance, it may be discovered that the number of units under a particular heading is greater than is called for, having regard to the capacity of the business or the extent of the operations likely to be undertaken, or the records may show that certain operations are being retarded by the continued use of inadequate equipment or an insufficiency of the right types. Experience has proved that changes can often be made to accelerate production, and that by rearranging the profit-earning assets it is possible to withdraw certain units and reduce costs. When rolling-mill plant, auxiliary equipment, attachments, accessories

and special devices are subjected to a physical control on the part of the management, it is certain that much more satisfactory results can be obtained than is possible when no attempt is made to exercise a check over the assets employed.

When the records take the form of cards kept in a drawer or cabinet, they are usually of convenient size and can be arranged in alphabetical or any other convenient sequence. At the head of each card, space can be reserved for the insertion of such particulars as the location and fitments, etc., and as installation expenses are usually written off against profits on a basis different from that applied to the asset itself, it is advisable to record details of the cost instead of lumping these together under one heading. Various styles of equipment card are in use at the present time and that shown may be regarded as a good general pattern ; it is capable of modification to satisfy any special requirement.

Various methods of computing and recording asset depreciation will be discussed in the next article which will also deal with the allocation of power and overhead charges.

£169,000 For Anodising Processes
Crown Payment to London Firm

A PAYMENT of £169,000 has been made by the Crown to Alumilite and Alzak, Ltd., for inventions and secret processes used during the war for the protection of aluminium and aluminium alloys particularly in the construction of aircraft. The award has been made without reference to the Royal Commission on Awards to Inventors and has been negotiated by Mr. A. J. Milne, managing director of the company.

Before the war the management caused the inventions of several people to be brought together under the auspices of the company and during the war made the collated information available to the Crown. Some 16 patents were made available in this way, primarily concerned with anodising processes and ancillary treatments. At the beginning of the war their application prevented a serious hold-up of production because they enabled industry to do without chromic acid which was imported.

Mond Nickel Fellowships 1950

THE Mond Nickel Fellowships Committee invites applications for five Fellowships of average value of £750 each for 1950. Fellowships will be awarded to selected candidates of British nationality with degree or equivalent qualifications to enable them to obtain wider experience and additional training in industrial establishments, at home or abroad, to make them more suitable for future employment in senior technical and administrative positions in British Metallurgical Industries. Each Fellowship will cover one full working year. Applicants will be required to state details of the programme they wish to carry out.

Particulars and application forms are available from :- The Secretary, Mond Nickel Fellowships Committee, 4, Grosvenor Gardens, London, S.W.1. Completed application forms are required by June 1st, 1950.

MR. E. BRUNSKILL (Mufulira Copper Mines, Ltd.) has been included in the Mond Nickel Fellowships Committee awards for 1949 to study the metallurgical practice in the mining, smelting and refining of non-ferrous metals in Africa, with special reference to copper and related metals.

The Corrosion of the Austenitic Stainless Steels

Part II—Pitting and Intergranular Corrosion

By G. T. Colegate, B.Sc., A.I.M.

Considerable progress has been made, during the last thirty years, in the development and application of the austenitic stainless steels. In a series of four articles, the author discusses many factors governing the corrosion of these steels, including the effect of alloying elements in addition to chromium and nickel, and of the corrosive media with which the material may come into contact in service.

IN the first article* of this series, following a general outline of the types of corrosive attack to which the austenitic stainless steels are subject, a detailed consideration of galvanic corrosion was presented. Two further forms of attack, pitting and intergranular corrosion are discussed in the following pages.

Pitting

It has already been observed that the pitting of stainless steels in many environments is one of the most serious forms of attack to which these alloys are subject. The causes of pitting are fairly well understood and it is thus possible, in some cases at least, to take certain steps to avoid this form of attack or to minimise the chance of its taking place.

In common with pitting in other metals, this form of attack is often rapid and, although the loss in weight of the metal as a whole may not be great, perforation or serious weakening may nevertheless be taking place at local spots.

Although pitting may occur in most metals under favourable conditions, stainless steel, in common with aluminium, is a metal in which, once a pit starts, penetration is likely to be particularly rapid. The reason for this is as follows ; pitting in metals is essentially an electrochemical phenomenon in which the pit starts at a point on the metal surface where, for some reason or other, the potential is less cathodic than over the rest of the surface. There are several possible causes for differences in potential over the surface of a metal ; for example, heterogeneity in the metal itself, presence of oxide scale on part of the surface, or differences in composition of the electrolyte in contact with different parts of the metal surface. In the case of stainless steel, the difference in potential is often set up at weak spots in the passive film. The presence of the film means that the metal is apparently more noble than it would otherwise be, so that, at any point where there is no passive film, the exposed metal will be anodic to the surrounding area of metal covered with the passive film. The difference in electrical potential between the active and the passive conditions of the austenitic stainless steels is considerable, much greater than the difference which normally exists between anodic and cathodic points on the surface of other metals, and amounts to half a volt or more. There is thus a relatively high voltage available, and a second factor which favours the attack, and is largely responsible for its speed, is the fact that the area of the anode is small compared with

that of the passive cathodic area surrounding it. Since the rate of corrosion is largely dependent on the amount of depolariser which can reach the cathode in a given time, the size of the cathode relative to the anode is obviously an important factor. There is, moreover, little or no chance of the anode becoming appreciably polarized and stifling the attack, since the products of corrosion in a chloride solution, for example, are chlorides, materials which break down any tendency to passivity and which, in any case, being in the ferrous condition, prevent access of oxygen to the bottom of the pit, thus preventing re-establishment of the protective film.

It will be evident, from what has been said above, that any factor which favours the local breakdown of passivity, at isolated points of the passive surface of the metal, will tend to encourage pitting. For example, any factor which encourages local depletion of oxygen in the medium may give rise to pitting. Among the best known of such factors are deposits on the surface of the metal. These act as an oxygen shield and, as pointed out elsewhere, the passivity of stainless steel is such that if it is to be maintained, there must be a constant supply of the agent causing it. Deposits of one sort or another are often a potent cause of pitting of stainless steel in sea water and may include marine growths, such as weeds or barnacles, sand, etc.

Corrosion at gaskets is particularly prevalent with stainless steel if the gaskets are allowed to become wet. Most gasket materials will cause the attack, even thin sheets of 18 : 8 stainless steel which are sometimes used for this purpose. About the most successful type of gasket appears to be a mixture of rubber solution with powdered metals, this being painted over the appropriate surfaces at the time of assembly. A modification of this consists of first applying the rubber solution and then sprinkling powdered metal over it, the excess being removed when dry. Not all metal powders may be used in such applications as some cause pitting of the stainless steel. Aluminium and copper are among the most active in this respect. Zinc, nickel or tin powder, on the other hand, may be used without difficulty.

Pitting is favoured by higher temperatures. This can be demonstrated in the laboratory and is also noticed in practice occasionally. If the temperature of a solution is raised sufficiently, however, the dissolved oxygen may be expelled and this in itself may be sufficient to reduce or even to prevent pitting entirely.

It has been shown that the pH of the solution in which the stainless steel is immersed also affects the rate of pitting, the greater the alkalinity, the less the liability
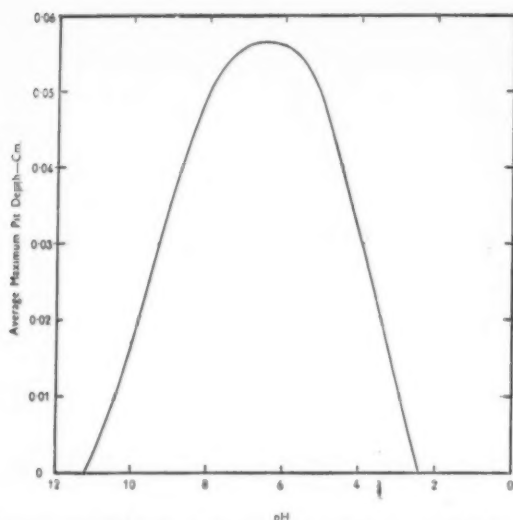
Fig. 1.—Relationship between pit depth and pH in 4% sodium chloride solution at 90° C.

to pitting, in general. For example, it has been reported that whereas pitting occasionally occurs in stainless steels in contact with brines in refrigeration plant of various types, if the alkalinity of the brine is increased by the addition of about 1% of sodium carbonate the resistance of the steel to this type of attack is greatly improved.

In sea water, on the other hand, the greatest tendency to pitting is noticed when the sea water is neutral. Both on the acid and the alkaline side of neutrality, the intensity of pitting and the maximum pit depth decrease, though in very acid sea waters having a pH of less than 3, the number of pits rapidly increases. The variation of maximum pit depth with the pH of a 4% sodium chloride solution is illustrated in Fig. 1 which summarises some work by Uhlig.[1]

Besides factors in the medium which favour pitting, there are also some associated with the metal itself. For example, heterogeneity in the microstructure caused by segregation of impurities or incorrect heat treatment may give rise to pitting. The holding of a stainless steel for a period at around 600° C. greatly increases its tendency to corrode. This is irrespective of any liability to intercrystalline corrosion, which such a heat treatment may bring about, and applies equally to titanium-modified steels. There is some evidence also that the presence of titanium and columbium, even in a correctly heat-treated steel, increases its tendency to pit compared with that of an unmodified steel. This applies mainly to exposure to such solutions as ferric chloride and not so markedly to exposure to sea water or other natural waters. Selenium additions are said to have the same effect.

Non-metallic inclusions may also provide nuclei for pitting, but practical experience indicates that such inclusions are not a serious cause of attack.

It has been claimed that cold-working of stainless steel increases its susceptibility to attack by pitting. This is only markedly so in the case of exposure to comparatively strong corrosives such as ferric chloride

1 " Corrosion Handbook " (Wiley), 1948, p. 170.
2 *Ibid*, p. 383.

solution. The same tendency is noticeable in sea wate and dilute chloride solutions but not to anything lik the same extent.

Pitting is less likely to occur on a highly-polishe metal than on one with a roughened surface, but it i frequently found that in the former case such pits a form are much deeper and deepen more rapidly tha those which occur in the latter case, when they are mor evenly distributed.

Oxygen may also be prevented from reaching th metal by contact with other metallic material, fo example, another piece of stainless steel.

A very common cause of pitting is the presence in th metal surface of a crack or fissure or else the crevice between the two pieces of metal fastened together with rivets or bolts. The cause in this instance is similar to that discussed above, namely, a difference in composition in the liquid in the crevice from that of the main bulk. The oxygen in the recess becomes exhausted and can only be replenished by diffusion of more oxygen from the bulk of the liquid. This diffusion into a very narrow crevice is an extremely slow process and it frequently happens that the deficiency is such as to enable a pit to start.

Even in the absence of any passivating film, a corrosion current can be set up as a result of differences in oxygen concentration in different parts of the electrolyte and appreciable currents caused to flow. The area where the oxygen concentration is least is the anode of the system. We thus have a cell which acts in the same direction as one caused by local breakdown of passivity resulting from a depleted oxygen supply.

If stainless steel is exposed to a rapidly moving solution, there is less risk of pitting than in a stagnant one for the main reason that, in such a solution, there is less risk of differences in oxygen concentration establishing themselves. This has been demonstrated experimentally by La Que[2] who describes the exposure of a stainless steel containing 17% chromium, 12% nickel and 2·4% molybdenum to stagnant sea water, in which case heavy fouling occurred, and also to water flowing at 4–5 ft./sec., when little or no fouling occurred. The loss in weight in grams from a specimen $12 \times 8 \times \frac{1}{4}$ in. exposed under stagnant conditions was 13, while under moving conditions it was zero. Similarly, the mean depth of pitting in the former case was 0·073 in. as against 0·002 in. in the latter. Moreover, the moving liquid carries away corrosion products which would otherwise accumulate in crevices and cracks. There is also less chance of marine growth taking place in moving sea water.

A further important factor in the production of pits in stainless steels is the presence of chloride. For this reason, pitting tends to be particularly prevalent in sea water. As mentioned elsewhere the chloride ion is particularly active in breaking down the passivity of stainless steels and it is not surprising, therefore, that its presence favours pitting.

Methods of Avoiding Pitting

From the few remarks made above, methods of avoiding pitting under practical conditions of exposure immediately suggest themselves, namely, maintenance of as uniformly oxidising conditions as possible, assisted by solution agitation ; avoidance of the presence of the chloride ion as far as possible ; increasing the pH of the medium. Recesses and pockets where stagnant liquid can accumulate should be avoided, this being largely a question of design.

mentioned previously, cathodic protection has also been shown to be effective in preventing pitting of stainless steel in sea water. Iron, aluminium or zinc may be used for coupling to the stainless steel and, provided that sufficient area of one of these metals is used, pitting can be completely eliminated.

Another method of preventing pitting when the stainless steel is part of a closed system is to add an inhibitor to the water. Chromates are the most effective substances for this purpose and will effectively prevent pitting in very low concentrations. They are, moreover, effective in the presence of chloride. Nitrate has also been used for this purpose but chromates are often preferable because, in a system containing other metals besides stainless steel, for example, carbon steels, they are equally suitable as corrosion inhibitors. Corrosion inhibitors are, of course, out of the question except where the system is a closed one or where the amount of liquid to be treated is relatively small; otherwise the treatment becomes uneconomical.

Intergranular Corrosion

Although the exact mechanism of the intergranular corrosion of stainless steels is still a matter of dispute, the conditions which produce the phenomenon are well understood and so are the remedial measures necessary to overcome the attack. It is, therefore, proposed merely to summarise these very briefly here for the sake of completeness.

The intergranular corrosion of these steels is a result of changes produced in their microstructure by heat treatment. Maximum corrosion resistance is obtained when the heat treatment has been such as to produce a single-phase structure. If, however, the steel has been held in a certain temperature range, between about 350° and 800°C., carbides of chromium are precipitated at the grain boundaries, resulting in depletion of chromium in the immediately adjacent metal. Whatever the exact mechanism of the attack, it appears to be associated with carbide precipitation, the lower the carbon content of the steel the less liable is it to this form of deterioration. The temperature at which the steel is held in the critical range and the length of time for which it is held at that temperature are also most important factors in determining the rate of attack. This information is conveniently summarised in a curve constructed by Bain, Aborn and Rutherford[3] which is reproduced as Fig. 2.

The intensity of attack depends, of course, on the exact conditions of exposure. Even in comparatively mild corrodents, attack can be very severe and virtually cause complete disintegration of the steel.

Prevention of Attack

Methods of minimising or avoiding this type of attack are, very briefly, as follows :—

1. To anneal the steel between about 1,050° and 1,100° C. followed by a rapid quench. Such treatment is not, of course, always practicable in fabricated parts. For example, it is by no means uncommon for a sensitive zone to be produced on either side of a weld and there are, obviously, many cases in which the annealing and quenching of welded structures is entirely out of the question.

2. To reduce the carbon content of the steel to a very low figure. There are now commercially available in

3 Trans. Amer. Soc. Steel Treating, 1933, 21, 481–509.
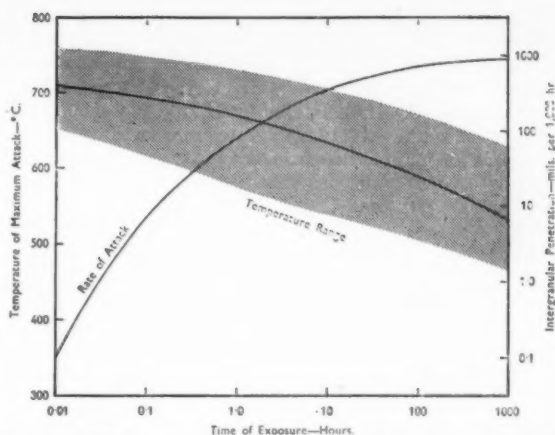
Fig. 2.—Effect of time and temperature on maximum sensitivity of low-carbon 18 : 8 stainless steel to intergranular attack.[3]

America low carbon stainless steels, of the straight 18 : 8 type and also of the molybdenum modified 18 : 8 type, which have a carbon content of 0·03% maximum. It is, as yet, too early to assess the value of this reduction of carbon, but results so far obtained are very promising.

It was at one time considered that, if the carbon content of this type of steel were kept below 0·07%, there was no risk of intercrystalline corrosion. Although it appears that such low-carbon steels are less susceptible to this type of corrosion, and that they are, in general, less severely attacked if it does occur, very serious cases are occasionally encountered.

3. To make additions of titanium or columbium. Several other metals have been considered as additions to stainless steel for this purpose, but most of them have either been a failure or had only a very limited success. Some of these are briefly considered below.

Titanium.—As long ago as 1933 it was discovered that small additions of titanium to the austenitic stainless steels conferred upon them immunity from intercrystalline corrosion. The amount required is usually expressed arbitrarily as a ratio to the carbon content of the alloy, a 5 : 1 ratio being normally used. If an amount of titanium is present below the critical amount required for complete suppression of intercrystalline attack, some degree of additional resistance is conferred upon the alloy as compared with the unmodified material.

The function of the titanium is to combine with the carbon in the steel to form a stable carbide, thus depriving the chromium of the opportunity of combining with the carbon itself and producing the depletion in chromium at the grain boundaries which gives rise to the poor corrosion resistance mentioned above. A good deal of work has been done on the question of the optimum titanium content, work that is much too extensive and detailed to discuss here.

Titanium has little effect on the general corrosion resistance of the austenitic stainless steels. It has been claimed by some that the presence of titanium reduces considerably the resistance of the steel to certain solutions, particularly nitric acid, but there seems to be little or no confirmation of this in practice.

Rosenberg and Dorr[4] carried out extensive work on the factors which influence the stabilisation of austenitic stainless steels and investigated 18 : 8 steels with both

high and low-carbon contents and with titanium : carbon ratios of 3·9–7·9. Specimens were examined in the cold-rolled condition and after eleven different annealing and stabilising treatments, the former varying from 980° to 1,080° C. The length of time for which the specimens were held at the temperatures in question was also varied and some of the samples were given a sensitising heat treatment for various times at temperatures of 450°, 550° and 650° C. The susceptibility to intergranular attack was judged by immersion for various periods in a copper-sulphate—sulphuric-acid mixture and the degree of attack was estimated in a number of ways. The conclusions reached by the investigation were that an increase in the ratio of titanium to carbon appreciably improved resistance to intercrystalline attack, the ratio which was necessary to give virtually complete immunity depending on the condition of the steel, but being independent of the carbon content. That is to say that titanium additions are equally effective in the higher carbon austenitic steels as they are in the lower carbon ones, provided that the ratio of titanium to carbon is the same in each case. Even in the presence of titanium it is found that a stabilising heat treatment is desirable. The authors claim that the ratio of 5 : 1 mentioned above is the minimum that should be used, and then only under the most favourable conditions of heat treatment. A ratio of 8 : 1 is considered to be more desirable.

Columbium.—The use of columbium as an addition agent to stainless steels to prevent intercrystalline attack is similar to that of titanium. It has not been used as long or as extensively as titanium, and a rather greater amount of it is necessary to ensure immunity from attack. Eight to ten times the carbon content is the figure generally accepted for the columbium content. Smaller quantities have the effect of reducing the range of temperature over which the steels become sensitive to intercrystalline attack.

There is little or no information available on the effect of columbium additions on the general corrosion resistance of the austenitic stainless steels but, under most practical conditions of exposure, it is impossible to detect any difference in the behaviour of the steels with and without columbium additions.

An investigation into the effect of columbium was carried out by Rosenberg and Dorr,[4] parallel with that described above on titanium. It was concluded that, as with titanium, increase in the columbium : carbon ratio improved resistance to intercrystalline corrosion. Unlike the case with titanium, however, no stabilising heat treatment is necessary when columbium is present in the steel. A columbium : carbon ratio of at least 10 : 1 is recommended to ensure more or less complete immunity from intercrystalline corrosion and a ratio of 12 : 1 is considered desirable.

A number of authorities take the view that columbium is much more satisfactory as a stabilising element in stainless steels than titanium.

Molybdenum.—This metal cannot be claimed to prevent intercrystalline attack on the austenitic stainless steels, although it does reduce the chances of such attack taking place and, if it does occur, it reduces its severity. Though this may be a useful asset when combined with the other beneficial effects which molybdenum additions have on the general corrosion resistance of stainless steels, it is not sufficiently pronounced to justify the use

of molybdenum alone as an element for suppressing the tendency to intercrystalline failure and cannot be compared with either titanium or columbium for this purpose.

Tungsten.—Tungsten has been regarded in the past as being capable of reducing the susceptibility of austenitic stainless steels to intergranular failure but there seems to be little justification for this view. The inclusion of a few per cent. of silicon in addition to the tungsten does in fact reduce the tendency to this type of attack but not to the extent that titanium and columbium do.

Other Metals.—It has been reported that tantalum additions to austenitic stainless steels render them insensitive to intergranular corrosion but, as far as the writer knows, there is no industrial application of tantalum as an addition element to these steels.

Similarly zirconium, vanadium and uranium additions have been claimed to reduce sensitivity to intercrystalline attack, but appear to offer little likelihood of commercial application.

Aberdeen Bridge

HEAD WRIGHTSON & CO., LTD. of Thornaby-on-Tees announce that their subsidiary Company, Head Wrightson Aldean, Ltd., has been entrusted with a contract for an aluminium opening bridge of the bascule type, at Victoria Dock, Aberdeen Harbour, by the Aberdeen Harbour Board.

The reduced weight of this aluminium bridge as compared with a steel structure will enable considerable savings to be made in the cost of adapting the existing dock wall to suit the new requirements. The clear opening is 70 ft. and the bridge will carry two lines of road traffic, or one of road and one of rail traffic.

This second aluminium alloy bascule bridge follows the success of the first bridge, which it slightly exceeds in size, constructed by Head Wrightson & Co., Ltd. in 1948, at Hendon Junction Dock, Sunderland, for the River Wear Commissioners. The decision is a further development in this and will again raise the question of constructing a roadway in aluminium alloy for vehicular traffic over the Forth Bridge.

Work on the Aberdeen Bridge, which will cost approximately £88,000, is to begin immediately and is expected to take some two years to complete.

New Paint Line-up

IN order to provide the shipping industry with a more efficient and more economical paint service, the marine interests of three long associated companies have been rationalised into one Marine Division. This operates within the orbit of British Paints, Ltd. and incorporates the marine interests of the British Anti-Fouling Composition and Paint Co., Ltd., and Messrs. J. & W. Wilson, Ltd. It is especially stressed that no change of Directors or Staff is contemplated in the re-arrangement.

This new Marine Division, which started operations on January 1st, will be entirely responsible for the supply of marine paints in all home and overseas markets under the universally-known brand names of " Torpedo," for large vessels, and " Little Ship " for smaller craft. Enquiries should be addressed to British Paints, Ltd., Marine Division, Royal Mail House, Leadenhall Street, London, E.C.3.

4 J. Res. Nat. Bureau Stands., 1948, 40, 321.

The British Non-ferrous Metals Research Association

Increased Laboratory Capacity

For many years now, the British Non-Ferrous Metals Research Association has been faced with the problem of " too many workers chasing too little space "—to borrow a phrase from the economic world. With the reconstruction of a laboratory block destroyed by bombing in 1940, this state of affairs has been remedied and, at the same time, equipment has been installed for the production of wrought materials for research purposes.

SINCE 1921, when the British Non-Ferrous Metals Research Association's activities were started in Birmingham on a very modest scale, with a membership of 20, its influence and importance in the non-ferrous industry have steadily grown, until to-day its members number just under 500 and the annual income from all sources has risen from £6,000 to just over £100,000. The Association first established its own laboratories in 1930, when a small factory block was rented in Regnart Buildings, Euston Street, London, N.W.1. The rapid expansion in membership and the accompanying increase in calls on the Association's services have necessitated the purchase and development of properties adjacent to the Euston Street site. Nevertheless, the increase in activities has generally kept in front of the expansion of the laboratory accommodation and when, in 1940, one of the laboratory blocks was destroyed by bombing, the accommodation difficulties were accentuated. This block has now been rebuilt, however, and with its reconstruction the unsatisfactory state of affairs which formerly existed has been remedied —for the time being at least. No doubt the expansion curve has not yet reached its peak but space is available for further expansion to the extent of some 30% of the 45,000 sq. ft. of floor space at present in use.

The reconstruction of the bombed block is the culminating point in the large expansion in accommodation, equipment and resources, which has taken place during the last four years, and the Laboratories were recently open for inspection of the new facilities.

The New Laboratory Block

The main addition to the Association's resources is a metal-working laboratory in the basement of the new building. The equipment installed is intended for the production of wrought materials for use in the Association's experimental work. In the past the Association has obtained these materials through the generous assistance of its members who have always been most willing to assist in this way, despite the inconvenience caused by working small batches of experimental materials in their production equipment. With the expansion of the Association's staff and facilities the demand for wrought materials has increased and simple equipment has therefore been installed to facilitate the preparation of the experimental materials.

The equipment includes a 500-ton down stroke hydraulic press, which can be used for extrusion or press forging. The speed of the main ram can be maintained constant at various pre-set speeds between the maximum (0·75 in. per second) and minimum (0·03 in. per second) rates, and the press is thus well suited for experimental extrusion on a semi-industrial scale. The press is designed to give an automatic forging stroke of any
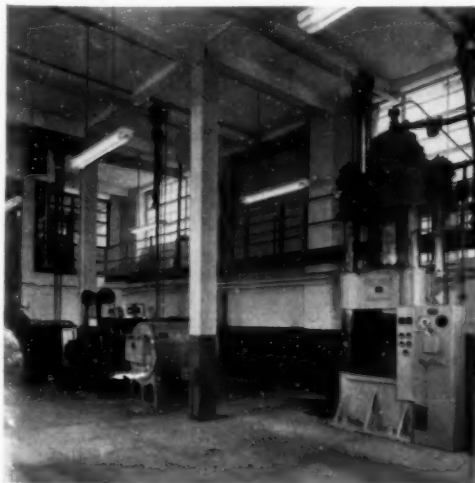
Metal working laboratory, showing the press, rolling mill and furnaces.
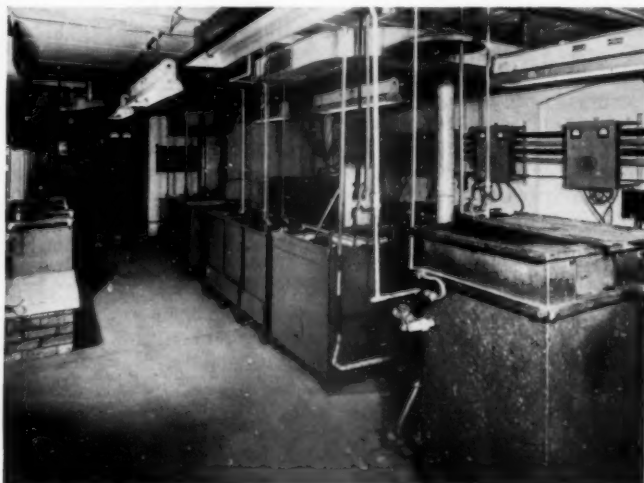
Electroplating laboratory, showing experimental plating vats and trichlorethylene degreasing plant.
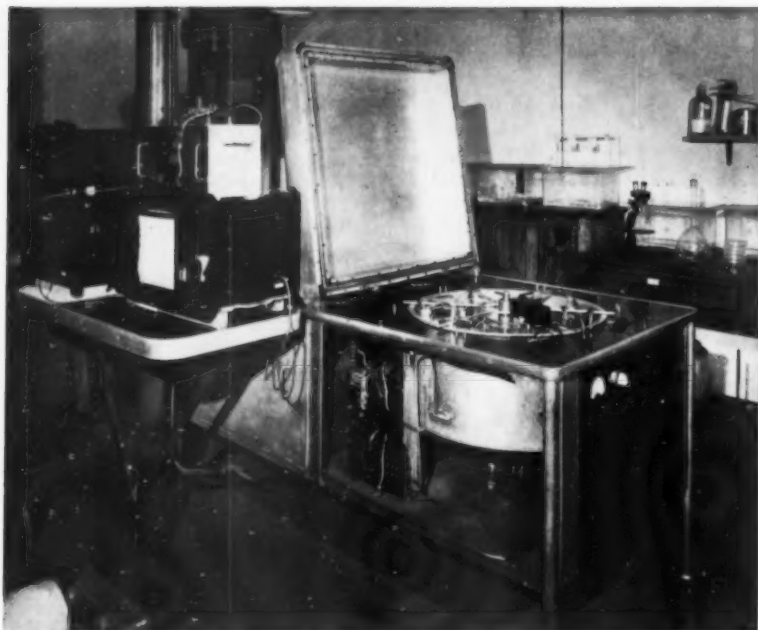
Apparatus for continuously measuring and recording electrochemical
potentials and corrosion currents during corrosion tests.

desired length from a few inches to 2 ft., the maximum
stroke of the press, and as the maximum daylight is
5 ft. and the minimum daylight 3 ft. there is ample
room for sub-press assemblies for extrusion or forging.

The second main item of equipment is a 12 in. $\times$
12 in. rolling mill, equipped with plain rolls for sheet and
strip production and with grooved rolls for the produc-
tion of bar material. Material too thick to be broken
down in the mill will be hot or cold forged in the 500-ton
press, and a 6 ft. long draw-bench is available to draw
the bar material produced in the mill down to rod and
wire (e.g., 22 S.W.G.) as required. Simple equipment for
straightening rod, sheet and strip is to be installed.

The laboratory is equipped with auxiliary items,
including two pre-heating and annealing furnaces with
6 ft. and 3 ft. long hearths, respectively, which are
designed to reach a maximum temperature of 1,000° C.
A small forced air circulation furnace has also been
installed with a maximum temperature of 600° C., and
this is particularly suitable for pre-heating and heat
treatment of light alloys. The equipment also includes
two smaller hydraulic presses of 50 and 20 tons, respec-
tively, suitable for powder metallurgy work on a small
scale. Finally, the equipment includes a number of
machine tools suitable for cutting up the wrought
materials made in the laboratory.

It is not intended that the equipment should be used
for the works application of laboratory developments ;
such work is, with members' co-operation, carried out
by the Liaison Department in members' works, employ-
ing industrial plant that is in general use.

The first floor of the new laboratory is devoted to the
General Metallurgy Section, whose activities had hitherto
been scattered in somewhat unsatisfactory places. On
the north side the metallographic and photographic
work are housed, and on the south side are the general
metallurgy and heat-treatment laboratories. These

provide room for apparatus used in
studying slag/metal and gas/metal
reactions, and the equipment include
a 7 kW. high-frequency furnace with
the flexibility necessary for such work
There is also a 10 kW. carbon-resisto
vacuum furnace of the Kroll type used
for example, for the melting of
titanium.

The second floor provides accom-
modation for the Director and Senior
Staff. This change has had the dual
effect of providing a central and
convenient location for the administra-
tive offices and of releasing existing
offices for absorption into the library
and physics departments where addi-
tional accommodation was urgently
needed.

The Association's Activities

This block cannot, of course, be
considered by itself, but only as part of
the laboratory accommodation as a
whole, which, in turn, is governed by
the constitution and aims of the
Association. The first concern is
research on the manufacture of non-
ferrous metals and alloys and their
selection for particular user purposes.
The industries served are numerous, with widely differing
plant, process and material problems, and it follows,
therefore, that it is necessary to have a balanced team of
first-class scientists capable of dealing with the many
different problems which arise. Researches are called
for on extraction problems involving the thermodynamics
of high-temperature reactions, on melting and casting
problems ranging from studies of gas-metal equilibria
to such practical problems as obtaining a good surface
on a casting. In the field of metal working and fabrica-
tion, problems range from fundamental structural
questions, on the one hand, to relatively simple deter-
minations such as power requirements in deformation on
the other. From the point of view of the user, the
demand includes information on such questions as
corrosion resistance, mechanical properties, etc., and the
development of alloys with improved properties in such
respects.

To meet these requirements, the Association is divided
into three departments, Research, Liaison and Technical
Service, and Information. The largest of these, both
from the point of view of staff employed and of the space
occupied, is the Research Department which is divided
into the following sections : Physics, Mechanical Testing,
General Metallurgy, Corrosion, Melting and Casting,
Chemistry and Metal Finishing.

The Research Department

The research programme is the responsibility of the
Research Board, a small Committee responsible to the
Council and constituted of scientists from industry
capable of advising on research policy. The Board is
advised by four Industry Committees composed again
of industrial scientists selected in this instance from the
copper, light alloy, lead and zinc industries, respectively,
whose duty it is to advise the Board on the researches
needed by the particular industries they represent. In

this way a programme is drawn up of industrial problems suitable for the facilities and staff of the Association and of importance to the different sections of industry. Many of these aim primarily at an elucidation of the phenomena underlying particular metallurgical processes, for it is only by a full understanding of these basic principles that progress can be achieved. A relatively small number of the researches are of an *ad hoc* type aiming to provide a solution to a specific problem.

The Physics Section, which occupies the ground floor of one of the Laboratory Blocks, is equipped with a range of spectrographic equipment which includes direct recording units of an experimental type. The main laboratory houses an electron microscope, X-ray crystallographic set and conductivity and damping capacity apparatus. Among the subjects on which investigations are being carried out are the metallurgical applications of the spectrograph and the electron microscope.

Apparatus for simultaneous measurements of temperatures at many points in the same casting or mould ; temperatures are recorded every tenth of a second. A complete record of the thermal gradients during cooling is thus obtained.

Although the usual facilities are available for the carrying out of the standard mechanical tests, a considerable proportion of the work of the Mechanical Testing Section is concerned with the properties of materials at elevated temperatures, including creep, fatigue and impact. The equipment includes 76 creep testing units suitable for use at temperatures up to 750° C. together with a battery of rotating-load fatigue testing machines for use over a similar temperature range. For the creep testing of lead and its alloys at 20° C., a constant-temperature room is available.
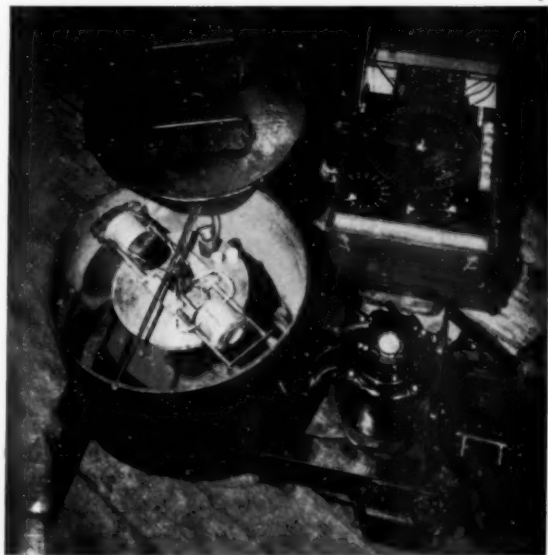
Centrifuge for the separation of primary constituents from semi-molten metals. This apparatus has been applied to the separation of primary particles responsible for grain refinement in aluminium alloys.

The work of the Association on the corrosion of non-ferrous metals and, in particular, that on the corrosion of condenser tubes is well known to those concerned with these problems. In this connection the Association can take a good deal of credit for freeing ships of the Royal Navy from condenser tube corrosion troubles. Quite recently the Corrosion Section have traced the cause of pitting of domestic cold water pipes (in certain supply waters) to carbonaceous films resulting from manufacturing operations. Contributions to the increasing knowledge of the effect of bacteria on the corrosion of metals have also been published in the last year or two. The Corrosion Laboratory is equipped for testing the corrosion of metals by moving sea water, for potential and current measurements, and for stress corrosion work.

The properties of the finished product, be it a casting, a forging or wrought bar and section material, owe much to the conditions of melting and casting, and for many years now the appreciation of the importance of these factors has been evident in the Association's research programme. The foundry, housed in the basement, is provided with a range of crucible furnaces and ancillary equipment, a 25 kVA. high-frequency melting furnace and facilities for sand moulding. Included in the researches in progress are the oxidation of molten zinc ; thermal gradients in castings ; the mechanism and prevention of metal/mould reactions in light alloys ; grain refinement of cast copper-base alloys ; and refractory linings for melting copper in Ajax-Wyatt furnaces.

The Metal Finishing Section, which is of comparatively recent origin, is mainly housed in the basement where equipment is available for the preparation and plating of articles on a considerable scale, and with a wide variety of different deposits. The adhesion of deposits on aluminium and magnesium ; the electropolishing of non-ferrous metals ; and the vitreous enamelling of aluminium are being investigated.

Whilst considerable effort is being made to get the results of investigations applied in industry, the need to maintain scientific capital is not forgotten. As Sir Ben Lockspeiser observed in his opening speech, "Science also has its gold reserves, and if they fall too low, technology loses the source of its sustenance." The Association accordingly has to keep a working balance between the effort it gives to acquiring new knowledge and that it gives to applying what is already known. One of the subjects on which the Association is breaking new ground is that of the properties of a hitherto neglected metal, titanium.

Other current investigations coming under the heading of General Metallurgy include the deep drawing of aluminium ; alloys for gas turbine heat exchangers ; recrystallisation of wrought aluminium alloys ; aluminium bronze welding ; and soldering and brazing.

The Liaison and Technical Service Department

The primary object of the Liaison Department is to help bridge the gap between the laboratory and industrial application. The Liaison staff, having satisfied themselves that a development is ready for trial on a production scale, bring it to the notice of the manufacturers concerned and secure their co-operation. The trials that result are reported to all members. Most of the time of the Liaison Department is, however, spent in helping members to use existing knowledge.

Information Department and Library

This Department serves both the members and the Association's staff. Its general function is to make available existing information on any subject which may be called for. Its operations are based on a library which is kept in first-class condition as regards non-ferrous metallurgy and related fields, and which has excellent contacts with a large variety of other libraries and organisations, so that any other literature can be obtained speedily. The current stock of the library amounts to some 5,000 books and bound volumes and 25,000 pamphlets.

The Information Department searches the literature for information on subjects of interest and in this way co-operates with the Liaison Department in the technical enquiry service. In addition many minor enquiries are answered directly by the Information Department. Translations of foreign papers are also produced (in 1949 some 30 translations were made). The Information Department also sees the Association's publications through the press, and acts as editor of the monthly Research Supplement, which is a confidential document supplied to members.

Design of a Large Vacuum Valve : Its Application to an Electric Furnace

By W. F. Atkins, M.I.E.D., A.M.I.P.E. and G. C. H. Jenkins

(Communication from the National Physical Laboratory)

A PROCESS has been developed by the Metallurgy Division of the National Physical Laboratory for the production of high purity iron on a 25-lb. scale, the last operation consisting of deoxidation of molten iron by hydrogen in a high-frequency furnace, which is installed in a large vacuum-tight tank. The equipment is evacuated by two large rotary pumps through an 8 in. (20·3 cm.) diameter pipe connected to the tank. In order to check that there are no leaks in the tank, and also to enable the introduction of hydrogen, it is necessary at times to isolate the tank from the pumps. To do this it was necessary to fit a valve in the 8-in. (20·3-cm.) diameter pipe which would fulfil the following requirements. In the first place, it should be completely vacuum tight in order to test for very small leaks in the tank and, secondly, it should not restrict the full pumping speed at low pressures. A further requirement was that the valve should be capable of being opened gradually so as to give a controlled rate of pumping over the whole range from fully opened to fully closed. This was essential because, during certain stages of the process, removal of hydrogen from the tank had to be adjusted so that the evolution of gas from the molten metal did not become so violent as to cause the loss of substantial quantities of metal by splashing from the crucible.

It was desirable that the valve should be introduced with the minimum alteration to the existing equipment and the most convenient position was in a right-angle elbow in the piping which allowed a range of movement of 12 in. (30·5 cm.) during the action of opening and closing of the valve.

A plug-type valve would have suffered from the disadvantage of being easily scored by particles of refractory material which might inadvertently be dragged over from the furnace lining—such particles are trapped between the valve and the pumps.

The valve, as constructed, is easy to operate, requires little or no attention and has given excellent service over a period of time. No appreciable diminution of pumping speed at low pressures has been observed since its installation.

Description of the Valve

The overall dimensions of the valve, including the flange and operating handle, are 24 in. (61 cm.) long and 12 in. (30·5 cm.) diameter. The major portion of the 12-in. travel during opening and closing is achieved by a straight line pull, the final pressure to bring the valve on to its seating being by means of a screw.

Altogether, three vacuum seals are incorporated. Referring to Fig. 1, the first seal is at W between the end plate T and the valve head V ; the second is between the cover plate A and the housing B ; whilst the third is at C between the housing B and the valve spindle G. These three, when the valve is closed, effectively seal the furnace from the pumps and from the atmosphere.

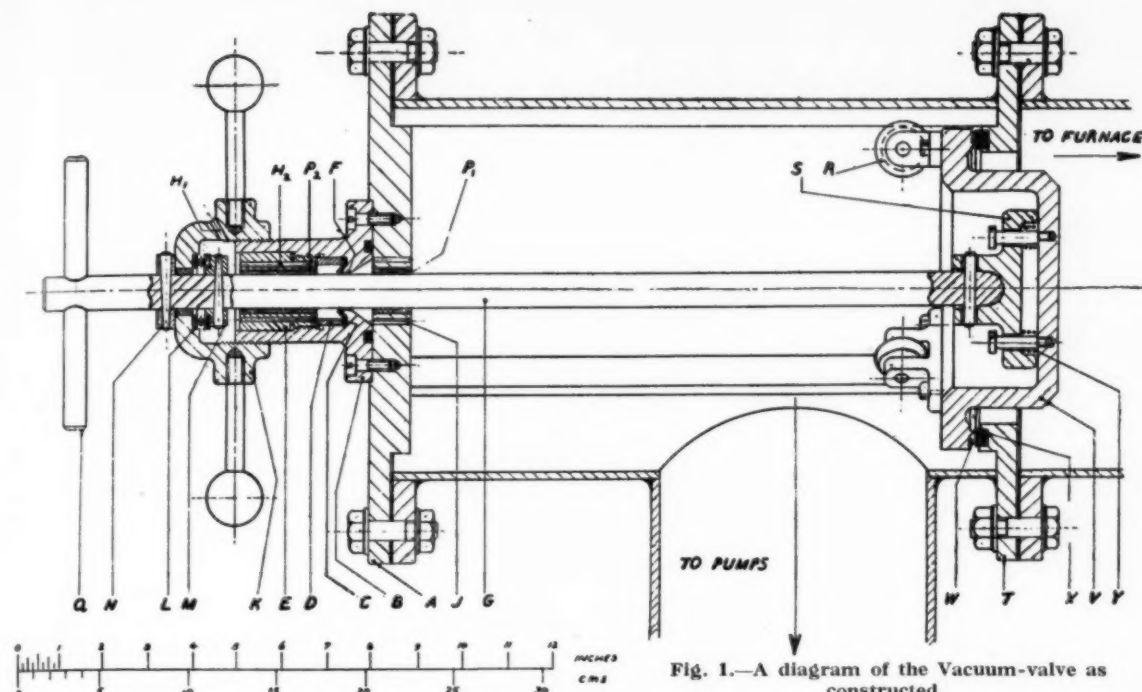It will be observed that the cover plate A is recessed

Fig. 1.—A diagram of the Vacuum-valve as constructed.

to locate the housing B, the base of which is grooved to accommodate a rubber ring. This provides the vacuum seal between the housing and the cover plate.

The vacuum seal between the housing B and the valve spindle G is of the Wilson type, consisting of a rubber ring C held on a 30° conical seating by the ring D and clamped by the threaded plug E. A rubber ring of $1\frac{1}{2}$ in. (3·8 cm.) diameter $\times$ $\frac{5}{8}$ in. (1·6 cm.) bore $\times$ $\frac{1}{16}$ in. (0·16 cm.) thick proved satisfactory for the valve spindle which has a diameter of $\frac{3}{4}$ in. (1·9 cm.).

Pressure exerted on the valve spindle by the inner edge of the rubber ring effects the vacuum seal. This pressure is reinforced by atmospheric pressure exerted on the ring through the holes H_1 and H_2, and by the vacuum acting through the holes J and the bore F. For the seal to function satisfactorily the bore F must be concentric with the valve spindle G, and the clearance between the two is of importance. A diametral clearance of 0·02 in. (0·5 mm.) proved satisfactory, and the necessary concentricity of the spindle and the bore is maintained by mounting the spindle in bearings P_1 and P_2, which are of the sintered bronze, oil-filled type, and are positioned one on either side of the seal.

Operation of the Valve

The cap K is free to rotate on the valve spindle between the thrust race L and the collar N and, when screwed on to housing B, exerts a forward thrust on the valve spindle through collar M. When opening the valve the pressure of the cap K against the collar N is relieved to a certain extent by the forces exerted by the rubber ring X and the springs Y; a thrust race between K and N is therefore unnecessary. Also, as soon as cap K becomes disengaged from the screw thread on housing B, it is possible to open the valve completely by a direct pull on handle Q.

The valve V has two pressure rings W to ensure a good vacuum seal and is positioned concentrically with

respect to the furnace inlet pipe by three wheels running on the rails provided. These rails are also disposed symmetrically with respect to the vertical axis of the pipe leading to the pump. The wheel R running on the uppermost rail which is on the vertical axis of the pipe is flanged to prevent rotation of the valve. The shape of the valve V is such that the connection of the spindle with the pressure pad S is made at its centre of gravity, thus eliminating any undesirable couple between the guide rails and the wheels.

The spigots on plates A and T locate these plates concentrically with the turned surfaces of the rails. It is important that the bore of plate T should not be less than the area of the pipe to the pumps, otherwise a reduction in pumping speed will result.

The pressure pad S is attached to the valve V by means of six spring-loaded shouldered screws.

After the required vacuum in the furnace is obtained, the valve is partially closed by pushing the spindle forward by the handle Q until the screwed cap K just contacts the external thread on the housing B; this brings the pressure rings on valve V in close proximity to the rubber ring X in plate T. The valve is then finally closed by rotating the screwed cap K with the handwheel provided.

At this stage the pumps are shut down and vented. A pressure difference is thus created by the high vacuum on the furnace side of the valve and atmospheric pressure on the pump side. If the atmospheric pressure on the valve is greater than that applied by the screwed cap K, the valve V is free to move forward against the rubber seating, and away from the pressure plate S, rendering any further adjustment of the screwed cap unnecessary.

The work described above has been carried out as part of the research programme of the National Physical Laboratory, and this paper is published by permission of the Director of the Laboratory.

Aluminium Alloys for Mine-shaft Equipment

New Cages at Gresford Colliery

Although a proportion of the increase in the amount of aluminium alloys used in general engineering may have been the result of the exceptional post-war demand for steel, there are numerous applications in which factors other than availability govern their adoption. One such field is that of mine-shaft equipment where lightness is expected to lead to increased efficiency.

THE new all aluminium-alloy mine cages, recently installed at the Gresford Colliery, are a notable instance of the growing use of aluminium alloys for mine-shaft equipment. Aluminium alloys were first used in this way a little over twenty years ago, for the liner plates of cages in a Westphalian salt mine. Since that time, they have shown their value—in many countries, and under severe conditions of service—in the skips and cages which bring salt, rock, ores and coal to the surface.

The advantages of aluminium alloys for such purposes are to be appreciated in relation to the generally accepted principle of higher weight for moderate cost, as applied to mine-shaft equipment. In cage-winding installations, weight reduction in cages and tubs may offer improvements in production otherwise only attainable by a complete reorganisation of the winding and haulage system of the whole working, and of coal-getting methods in general.

For various reasons, lower cage weight can bring about a cumulative reduction of forces, weights and power consumption within the whole winding system. The possible advantages to be obtained from reduced tare-weight/pay-load ratios are briefly as follows :—

1. Increasing the safety factor and easing maintenance, load and power requirements on existing winding gear.
2. Improving output, by increasing pay-loads or winding speeds with existing winding gear.
3. Enabling greater depths to be wound without the need for installing heavier gear at the pit head.

The cages at Gresford, completed in August, 1949, and now in full operation in the 776-ft. deep main winding shaft, are the first, with a main structure entirely of aluminium alloy, to be employed in British mines. This step in re-equipment was taken by the North Wales No. 5 Area of the National Coal Board (North Western Division), under Mr. G. Nicholls—Area General Manager, in conjunction with Mr. H. Watson-Jones—Divisional Chief Mechanical and Electrical Engineer, and Mr. H. E. Dudley—Area Chief Mechanical Engineer.

The design and construction was in the hands of W. G. Allen & Sons (Tipton), Ltd., in collaboration with T.I. Aluminium, Ltd.—suppliers of the aluminium alloy material, and with Drynamels, Ltd.—suppliers of the paint system. Advice on the most suitable alloys for the purpose was obtained from the Aluminium Development Association.

To obtain the best results in the substitution of aluminium alloys for other materials it is usually necessary to change the design of stressed parts. In the new cages the forged vertical bar and hoop construction of the original steel cages has been replaced by a fully braced structure using aluminium alloy B.S. 1161 standard sections, flat bar and plate, with riveted joints throughout. The size and general layout conform to the unaltered requirements of the shaft-winding gear—viz.: Overall height—24 ft.; overall length—14 ft. 5 in.; and overall width—4 ft. 6 in. Each cage has three decks and carries nine coal tubs weighing 18½ cwt. each, fully laden—3 tubs per deck—giving a total carried load of some 8 tons 6½ cwt. The unladen weight of each cage is 4 tons 7½ cwt., as compared with a weight of 9 tons for a steel cage.

Framework during construction.
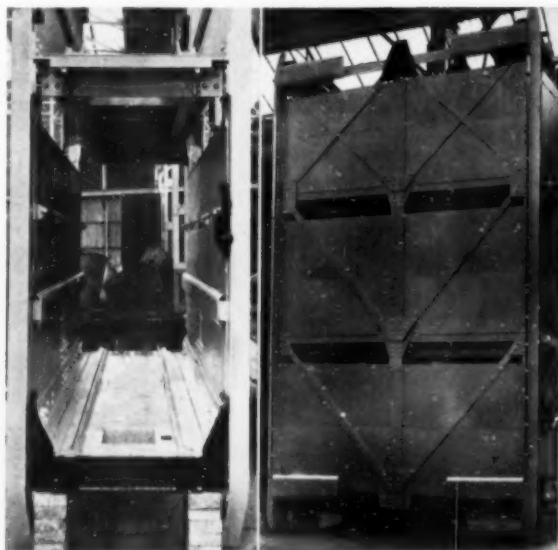
End view—bottom deck.   Side view—doors in position.

From the alloys supplied by T.I. Aluminium, Ltd., T.I. Alloys 01 and 04, conforming with British Standard (General Engineering Series) Specification H. 14, were selected for the main structure. Certain components with special properties were made in T.I. alloys 05 (B.S.S. N.6), 66 (B.S.S. H.15) and 77 (D.T.D. 683). Most of the structural members, gussets and cleats, called for material in straight lengths or flat plates—jig drilled and tack bolted on assembly. Certain components required forming operations—cold and hot—followed by heat treatment to develop the full mechanical properties.

The paint system, on which the operation of mineshaft equipment makes severe demand, was devised as a result of joint consideration of the problem by the three firms involved in the project. In the first stage—

carried out before assembly—all the finished members were coated with a special primer which etches the material and forms a thin coating with high adhesive and protective qualities. A coating of zinc chromate paste was also used where steel fittings were secured to the aluminium material. The completed structure was coated with red oxide zinc chrome primer followed by a finishing coat of aluminium paint. This "three-coat" treatment has been developed to resist the heaviest wear and tear.

The performances of these cages will be a valuable guide to the overall reorganisation planned by the National Coal Board on the basis of the Reid Report, in which emphasis has been laid on the need for higher efficiency in methods of coal haulage. Light alloys may also be expected to help in other aspects of this problem.

Motor Industry's Largest Baling Press
Labour and Transport Charges Reduced

Dealing with scrap metals has always been a problem in small and large works, particularly the latter, and the need for its constant removal for re-use is important not only for economic reasons but to prevent congestion and interference with production. The solution of the problem varies with the type of works, its products and the amount and character of scrap to be disposed of; a solution by a works with about 1,300 tons of scrap per month is described.

A general view of the press showing scrap being tipped from the weighing hopper into the container. Part of the cover is seen in the foreground and a completed bale stands on the roller conveyor.

A POWERFUL hydraulic scrap metal baling press just installed at Vauxhall Motors' Luton factory is saving the firm nearly £200 per week in reduced labour and transport charges. The press, designed and built by The Loewy Engineering Co., Ltd., of London, is the largest in the British motor industry and among the most powerful in the world. The press has doubled Vauxhall's baled scrap capacity—yet it needs fewer men to operate than the smaller presses it supersedes, and the bales of scrap it produces are more compact, saving valuable space in railway wagons and cutting down handling costs.

In the production of Vauxhall cars and Bedford trucks the sheet metal left as "waste" in the press shop after

the various parts have been pressed and trimmed to shape amounts to over 1,300 tons a month. This scrap is pressed into bales and then sent by rail to the steel works for remelting.

Before the new press came into service, the limited capacity of the old scrap baling plant was a major problem. Much of the scrap which could have been baled with suitable plant had to be loaded into railway wagons in loose form, thus increasing transport costs. Moreover, scrap metal buyers pay more for baled scrap than loose metal because it is more suitable for charging the steelmaking furnaces.

The new baling plant occupies an entire building 75 ft. long, 34 ft. wide and 34 ft. high from foundations to

roof. Operating 16 hours a day, it bales well over a thousand tons of scrap each month at the rate of one bale every 3 minutes—but this rate can be increased if necessary. The bales measure 18 in. wide × 15 in. high, and vary in length from 32–48 in. The weight of each bale varies between 5 cwt. and 10 cwt. depending on the sort of scrap handled—which includes steel sheet up to $\frac{1}{4}$ in. thick.

Scrap is conveyed by run-about trucks from the press shop and tipped into a hydraulically-operated loading bin. This bin has a weighing device so that bales of a controlled weight can be produced. Up to 10 cwt. can be handled at one time, but the normal load is about 7 cwt. When the right weight is reached, the hopper is hydraulically raised and turned over so that its contents are discharged into the large container of the press.

At the turn of a wheel at the control desk, a massive lid slides over the filled container. Only after the lid is closed can the three pressing rams be operated in the pre-determined sequence—one ram forward, one ram sideways and one ram upwards, and only after the ram movements have compressed the scrap into a bale can the cover be moved back to permit the bale to be ejected from the press. As it is ejected by the press the bale is pushed by the cover on to a gravity roller conveyor for loading straight on to a lorry for transport to the rail siding. A Bedford-Scammell articulated six-wheeler operating with a fleet of three trailers carries 24 bales at a time to the railway siding. Far fewer railway wagons are used than before as a 13-ton wagon which will only hold 7 tons of loose scrap will easily accommodate its full load of 62 5-cwt. bales. A month's output of scrap therefore needs 26 fewer wagons than before.

Total cost of the new equipment, including foundation, building work, etc., is in the region of £28,000. Efficiency, however, has so much improved that according to Vauxhall's forecast the capital outlay will have been fully recovered by 1952.

Technical Details

The press plant consists of the baling press, loading and conveying equipment, and an hydraulic air accumulator with high-pressure pump.

The design and layout of the installation ensures economical transport of scrap and bales to and from the press and facilitates maintenance requirements.

The press consists basically of the container and three working rams. The container body is fabricated of strongly-ribbed steel walls securely bolted together and lined with renewable hard steel wearing plates. The plates are slotted longitudinally to prevent small pieces of scrap being lodged between them and the ram heads to eliminate any jamming. The container size is 8 ft. 11 in. × 3 ft. 9 in. × 4 ft. 4 in. deep. This allows the loading of bulky and long pieces of scrap. It is closed by an hydraulically-operated sliding cover, which runs in four self-aligning wedge pieces. In its closed position, therefore, the cover is firmly clamped down on the container.

The three rams are arranged to work in sequence, one forward, one sideways and one upwards, so that a compact bale is produced. After completing their pressing movements, the forward and side rams are locked in position and the pressing cycle is completed by the third ram moving upwards. The upward ram is also used for ejecting the bale from the container. The finished bale is tightly pressed and free from fins and jags.

By the side of the press is an hydraulically-operated loading bin with a weighing device fitted with a large dial. A distinctive feature of the weighing device is an arrangement by which the bin is automatically detached from the lifting levers, so that the true weight of the scrap can be accurately controlled. In its loading position the bin is at floor level, so that the run-about trucks which bring the material from the press shop can tip it into the bin with the minimum of handling. This arrangement, and the fact that the control of the bin movements and press movements are centralised, facilitates loading with the desired weight of scrap. Loading takes place whilst the previous load is being compressed in the press.

The press is driven by a Loewy pistonless air hydraulic accumulator of 100 gallons useful water capacity, working at 2 tons/sq. in. working pressure, and a total capacity of 140 cu. ft. The pressure water is fed into the accumulator by a Loewy horizontal three-throw pressure pump of 34 gallons per minute capacity, driven by a 150-h.p. motor. The accumulator consists of seamless drawn steel air and water bottles. When the plant is being put into operation the air bottles are filled by means of a small compressor and as the loss of air is very small, even over a lengthy period, only short runs of the compressor are required to replenish such losses.

The pressure pump is of the totally enclosed type. It is equipped with a forced feed lubrication system, and suction and delivery valves have seats of stainless steel. An electric motor drives the pump through a high efficiency speed reducing unit, with flexible couplings on the high and low speed shafts.

When the pump has filled the water bottles up to the highest level, the by-pass valve automatically by-passes the water delivery into a suction tank, and when water is drawn from the bottles the by-pass apparatus switches the water delivery back until the desired water level is again reached. Electrically controlled low-level valves shut off the water supply from the accumulator before the lowest water level is reached, so that no air goes into the delivery pipes leading to the press. In addition, an electric safety device and electrically-controlled acoustic signals come into operation to inform the operator when the highest pressure or the lowest water level has been reached.

The accumulator drive has the advantage over direct pump drive—particularly for this press with a long container and, therefore, a long pressing stroke—as there is always an adequate water supply provided by a comparatively small pump. When baling long pieces the speed of pressing can be as fast as the resistance of the material will allow, and the speed of the pressing strokes is not limited by the pump capacity.

All movements of the press are actuated and controlled from a control desk in the required and pre-determined sequence. The control desk is equipped with pressure gauges, showing the working pressure of each ram, and signal lamps to indicate the completion of each stroke. One man has complete control of all operations for loading, pressing and discharging of the material.

Locomotive Boiler Tubes for India

CONTRACTS for 470,000 ft. of locomotive boiler and flue tubes, placed by the Indian Government, are now being completed by the Tube Investments' subsidiaries, Howell & Co., Ltd., and Tubes, Ltd.

Domestic Copper Piping

Causes of Pitting Corrosion

*Recent work has revealed the main cause of pitting in copper piping used for cold water,
and this statement by the British Non-Ferrous Metals Research Association is given for
the guidance of manufacturers and users.*

THE British Non-Ferrous Metals Research Association
has, for several years, been studying the behaviour
of copper in supply waters, with particular reference to
the pitting type of corrosion which occurs occasionally
in certain districts, especially in cold water pipes. The
number of pipes affected is extremely small compared
with the total quantity in service. Recent work has
revealed the main cause of this type of corrosion in cold
water pipes, and the following statement is made for the
guidance of manufacturers and users of housing tubes.

This statement is based upon the Association's own
experimental work, coupled with a detailed survey of
available service information ; it is made with the
approval of the committee advising the Association on
its research on the corrosion of copper pipes in supply
waters.

1. There is no evidence to establish the superior
corrosion resistance of either of the two grades of
copper allowed by the British Standards for Light
Gauge Copper Tubes for water, gas and sanitation
(B.S. 659 : 1944) and for Copper Tubes to be buried
underground (B.S. 1386 : 1947). The information
available suggests that the two materials
phosphorus - deoxidised non - arsenical copper
(B.S. 1172 : 1944) and phosphorus-deoxidised
arsenical copper (B.S. 1174 : 1944)—are equally
satisfactory in service provided they are both free
from the carbonaceous films referred to below.

2. There is strong evidence that the pitting corrosion of
copper cold water pipes sometimes experienced in
certain waters is usually due to the presence of
carbonaceous films which may be produced inside
the tubes by some manufacturing conditions.

3. Pitting corrosion of copper pipes does not occur in
the majority of supply waters in this country, even
if carbonaceous films are present.

4. There is as yet no clear evidence concerning the
effect of carbonaceous or other films, originally
present, on the serviceability of copper tubes in hot
water systems.

A brief review of the evidence on which this statement
is based is given below.

Influence of Character of Water

Two types of corrosion of copper water pipes have
been observed.

"Green staining."—Green staining in baths, wash
basins, etc., is occasionally experienced with waters
containing a high proportion of free carbon dioxide. In
such cases slight uniform corrosion occurs which does
not appreciably reduce the useful life of the pipe. The
cupro-solvency test included in the usual water
engineers' analysis of a supply water indicates whether
the water is liable to give green staining if used with
copper tubes.

Pitting corrosion.—This may cause failure of copper
pipes by pinhole leaks, but has only been known to occur
in a small number of supply waters in this country. It

may occur in either hot or cold pipes but rarely in both
with the same water. Hot water pitting appears to be
confined to certain soft moorland waters whereas cold
water pitting, which may cause failure in quite short
periods, is mainly confined to moderately hard bore hole
waters although its incidence bears no apparent relation-
ship to the usual water analyses. Recent research has,
however, shown that most supply waters contain a
natural inhibitor which prevents pitting ; trouble is
experienced only in waters containing no inhibitor.
The inhibitor has not yet been identified and may be
different in different waters.

Effect of Composition of Tube

An analysis of 104 failed tubes reported to the Associa-
tion during the last 10 years shows that seven were of
tough pitch arsenical copper, 33 of tough pitch non-
arsenical copper, 32 of phosphorus-deoxidised arsenical
copper and 32 of phosphorus-deoxidised non-arsenical
copper.*

These figures do not give a measure of the probability
of failure occurring with a given type of copper. Thus,
while at first sight tough pitch arsenical copper appears
to be the most satisfactory material, this conclusion is
not justified since it is known that comparatively few
tough pitch arsenical tubes have been installed. Owing
to the impossibility of obtaining data on the numbers of
tubes of each of the types in use in the areas where
failures have been observed, the relative merits of the
various materials cannot be established by this analysis
of failures. It is also impossible because, as is shown
later, copper pipes may differ in respects more important
than composition.

Two cases have been investigated in which both
arsenical and non-arsenical deoxidised copper tubes
were in the same hot water installation and in which
failure occurred in a non-arsenical copper tube. In the
first case, failure occurred in the horizontal flow pipe—
the position in which failures usually occur in hot water
systems—and the arsenical return pipe was slightly
pitted. In the second case, the arsenical copper pipes
were unattacked, but it is not definitely known what
positions were occupied by the two types of copper.

Effect of Carbonaceous Films

Following the observation of carbonaceous films in a
number of pitted tubes, determinations were made of

OCCURRENCE OF CARBON IN COPPER WATER PIPES.

Weight of Carbon, mgms./sq. dm.†	Cold water pipes		Hot water pipes	
	Pitted	Unpitted	Pitted	Unpitted
Less than 1·0	7	12	3	1
1·0–1·9	10	1	8	0
More than 1·9	63	0	13	3

* The tough pitch copper pipes would not now conform to B.S. 659 : 1944 or
B.S. 1386 : 1947, both of which specify phosphorous-deoxidised copper.

† 1 sq. dm. is approximately equal to the internal area of a tube 6¼ in. long by
⅞ in. nominal bore.

the amount of carbon in the scale on the above-mentioned 104 failed tubes and on 17 sound tubes taken from service in areas where pitting has occurred. A close correlation was found between the amount of carbon present and the incidence of pitting in cold water, as shown in the table on page 271.

Out of 80 cold water failures, 63 had more than 1·9 mgm. of carbon per sq. dm. and only seven had less than 1·0 mgm./sq. dm., while out of 13 unpitted cold water pipes none had more than 1·9 mgm./sq. dm. and all but one had less than 1·0 mgm./sq. dm. The correlation between the presence of carbonaceous films and the occurrence of pitting in hot water is less definite.

These figures indicate that carbonaceous films were responsible for at least three-quarters of the cold water pipe failures examined. Some at least of the remaining failures are thought to be due to a certain type of oxide scale formed in the tubes during manufacture. It is believed that the carbonaceous films are formed during manufacture by decomposition of remnants of drawing lubricant when a non-oxidising atmosphere exists in the tube during annealing. The only type of oxide scale with which pitting has been associated is a closely adherent, shiny, reddish layer.

Both carbonaceous films and the oxide scales just described lead to pitting, in waters which do not contain the inhibitor, by providing large cathodic areas which localise attack at pores in the film or scale.

There is ample evidence that copper water pipes free from such carbonaceous films and oxide scales are satisfactory even in uninhibited waters.

Failures of copper water pipes are encountered in relatively few areas in this country, yet it is almost certain that some of the pipes installed in other areas will have contained carbonaceous films or oxide scales of the above type. It can be concluded that the presence of such films or scales will initiate pitting and lead to failure only in supply waters which do not contain the inhibitor referred to earlier.

Staff Changes and Appointments

Mr. R. D. Hume and Mr. R. A. Miller have been appointed Joint General Managers of Foundry Services, Ltd. Mr. Miller was formerly Technical Manager in charge of home sales and all technical problems ; whilst Mr. Hume was General Works Manager and also handled export sales. Under the new arrangement Mr. Hume will still be mainly concerned with production, and Mr. Miller with the foundry problems of the Company's clients, but greater unification and co-operation will now be possible.

Mr. E. H. Goater, formerly Sheffield area representative for Messrs. Wm. Jessop & Sons, Ltd., and their associates, Messrs. J. J. Saville & Co., Ltd., Sheffield steelmakers, has now taken over the representation of these companies in the Yorkshire (Leeds) area. His successor for the Sheffield area is Mr. A. W. Pryor, 25, Castlewood Road, Fulwood, Sheffield, 10, Telephone No. 33462. These two companies have also appointed a new representative for the South Wales area, Mr. R. S. Lloyd, 46, Bryn Road, Brynmill, Swansea, Glamorgan.

Mr. A. J. Harby has been appointed Traffic Manager of the Steel Division of The Steel Company of Wales, Ltd. in place of Mr. W. B. Draper, who has left to take up a position with Brush Engineering Co., Ltd.

Mr. E. Millington, Steelworks Manager at the Panteg Works of Messrs. Richard Thomas & Baldwins, Ltd., has been appointed Assistant General Manager.

Mr. John Hitchcock, B.Sc., who since 1946 has been Personal Assistant to Mr. Ivon A. Bailey, Managing Director, of Henry Wiggin & Co., Ltd., has now been appointed to the newly created post of Assistant Managing Director. Mr. Hitchcock was formerly a member of the Mond Nickel Company's Development and Research Department staff.

Mr. G. P. Tinker has been appointed as Managing Director of Birlec, Ltd. in succession to the late Mr. A. G. Lobley. Mr. Tinker, a Director of Birlec, Ltd. since 1946, brings to his post an intimate knowledge and experience of the business, having been a member of the executive staff of the Company since its formation in 1927.

Mr. E. S. Waddington, F.S.E., M.Inst.W., A.M.I.E. (s.a.) Associate I.E.E., of the Industrial Department of Philips Electrical, Ltd., has been appointed to the Executive Committee of the International Welding Congress of 1951.

Mr. E. Elliott has been appointed Information Officer to the Aluminium Development Association. Mr. Elliott has served for some time as Liaison Officer and Secretary to the Association's technical committees.

Mr. N. U. Bremner has left Messrs. Guest, Keen & Baldwins Iron and Steel Co., Ltd. to take up an appointment with Messrs. Richard Thomas & Baldwins, Ltd.

Mr. Luther Griffiths has been appointed Chief Engineer of the Steel Division of The Steel Company of Wales, Ltd. He will be in charge of the Engineering Department of Port Talbot, Margam and Abbey Works and Cornelly Quarry.

Mr. T. H. Wilson has been appointed Staff Foreman of the arc furnace melting department at the Ford Motor Co., Ltd., Dagenham, Essex.

Obituary

It is with deep regret that we have to announce the death of Mr. A. P. Newall, J.P., M.I.Mech.E., Chairman and founder of A. P. Newall & Co., Ltd., Glasgow, who died at Perryston, Ayr, in his seventieth year, on Saturday, February 18th, 1950.

We regret also to report the death, on February 20th, 1950, of Mr. Ernest B. Muscroft, of 53, Woodville Gardens, London, W.5, after a short illness.

Mr. Muscroft, who was 63 years of age, was a well-known and highly respected personality in the Iron and Steel Trade. He commenced his business career with Thos. W. Ward, Ltd., at Sheffield, in 1900, and in 1923 took over control of the Company's Scrap Iron and Steel interests in South Wales. In 1937 he undertook special duties with the British Iron and Steel Corporation, Ltd., in connection with Scrap supplies to the steel industry, which brought him into close contact with both the consuming and supply side of the industry, and he remained very active in this connection until his death. He had a wide experience of all matters relating to the industry, and his loss will be severely felt.

Recent Developments in Materials Tools and Equipment

The New G.R. " Ferroclad " Metal-cased Brick

CHEMICALLY-BONDED (unfired) basic bricks are being used in basic open-hearth furnaces in ever-increasing quantities and their application has become standard practice in many firms. During the past few years, numerous trials have established beyond doubt that chemically-bonded bricks give longer life if jointed with metal plates. The fusion of the metal plate binds the brickwork together to prevent spalling and joint erosion. General Refractories, Ltd., have recently commenced the manufacture of completely metal-cased bricks, under British Patent No. 546220. These bricks are cased on all four sides and are made in three qualities :—

" Ferroclad 10 "—a chemically-bonded chrome brick.

" Ferroclad 30 "—a chemically-bonded chrome-magnesite brick, and

" Ferroclad 70 "—a chemically-bonded magnesite-chrome brick.

These G.R. " Ferroclad " bricks are made in normal standard sizes and are recommended for use in front walls, back walls and ends of basic open-hearth furnaces ; walls and ends of copper reverberatory furnaces ; in certain cases for the side walls of electric furnaces ; and for certain positions in soaking pits.

The chief advantages of " Ferroclad " are as follows :

The metal case, being bonded to the brick during manufacture, adheres rigidly to the brick and avoids all looseness between brick and plate ; avoids the necessity to stock or handle loose plates and the risk of accidents due to handling loose plates ; prevents damage to bricks in transit ; requires no jointing material ; enables easier and quicker installation and considerable saving of labour ; eliminates spalling ; reduces or eliminates air infiltration ; suitable for hot repair work ; size and shape are regular ; and gives longer life.

Full technical information is available to all interested in this important basic refractory development from General Refractories, Ltd., Genefax House, Sheffield, 10.

Immersion Heaters for Acid Liquors

THERE are many instances where the heating of acid liquors, such as pickling and plating solutions, can, with advantage, be effected electrically. It is, however, essential that no contamination of the solution shall occur as a result of attack on the heater envelope. This requirement calls for an envelope which is acid proof, in addition to being a good insulator and unaffected by severe thermal shock.

Vitreosil (pure fused silica) is ideal for the purpose as it is completely unaffected by all acids, except hydrofluoric and, at high temperatures, phosphoric acid. It is also one of the best electrical insulators and is highly resistant to severe temperature changes. The efficiency of such heaters is very high but, as with any form of internal heating, the full benefit is only obtained if the vessel is well lagged and covered as far as possible.

The latest models of Vitreosil immersion heaters are an improvement on those previously supplied, the following modifications having been made :—

(a) The heater size, for a given rating, has been reduced, thus giving a greater heat transfer per unit area.

(b) The heaters are fitted with a rubber seal and the minimum immersion depth is marked by a safety-line round the envelope. The rubber seal, together with the attached rubber-covered leads, results in effective insulation.

Vitreosil immersion heater in position in acid vat.

(c) The outer surface of the envelope is glazed, thus giving more resistance to chemical attack and a surface more readily cleaned.

(d) An earth-wire has been incorporated within the envelope, giving additional protection.

The standard range includes heaters of $0 \cdot 25, 0 \cdot 5$, $1 \cdot 0$, and $2 \cdot 0$ kw. rating, suitable for use on a 110-volt supply, and of $0 \cdot 25$, $0 \cdot 5$, $1 \cdot 0$, $2 \cdot 0$, $3 \cdot 0$ and $5 \cdot 0$ kw. rating for use on voltages in the range 200-250 volt, as specified by the customer. From the 1 kw. size upwards, there is a range of overall lengths, although the immersion depth is the same, for a specified rating, regardless of the overall length. Prices range from £1 4s. 6d. for the $0 \cdot 25$ kw. model to £10 2s. 6d. for the largest of the 5 kw. models.

The Thermal Syndicate Ltd. Wallsend-on-Tyne.

Self-winding Electric Cable Reels
Notable Design for Machine Tools

IN operating machine tools, and much other electrically driven equipment involving position movement, including adjustable portable lighting sets, it is necessary to operate with loose cable for the supply of the current.

The general practice, however, of using long lengths of trailing cable is most objectionable. Apart from the dangers of tripping over it, entanglement with moving parts of machine tools and other machinery, and damage because of being run over by trucks and barrows, the
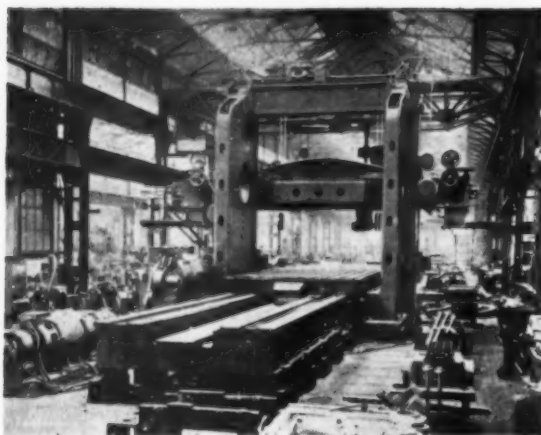
Craven Heavy Duty planing machine fitted with Wayne patent self-winding electric cable reels.

cable is deteriorated by contact with water, oil and other products, and there is serious risk of electric shock and short circuit.

These troubles can be avoided by using the " Wayne " self-winding electric cable reel, a production of Power House Components, Ltd., of Nottingham, under the direction of David Rushworth, and sold under the registered trade mark " P.H.C." Essentially this consists of a compact rotating metal reel on which the cable winds or unwinds, having a spring mechanism in an enclosed casing on a similar principle to that of the spring-roller blind, that is, the cable is paid out against the operation of a spring which always maintains a suitable tension without any undue slack or sag, and when the latter decreases the cable automatically rewinds with the action of the spring.

These self-winding cables are available in nine standard types according to the length and diameter of the cable used, that is the " Baby," " Standard," " Major 10/3," " Major 12/3," " Super 12/515," " Super 15/6," " Super 19/4," " Goliath " and " Mammoth." The latter, being the largest, differs from the other designs in that the return action because of the great weight of the cable used, $2\frac{1}{4}$–3 in. diameter, is sometimes carried out by hand-operation wind instead of a spring.

A typical example is the " Craven " heavy-duty planing machine, $12 \times 12 \times 40$ in. stroke. Three " Wayne " super reels are used in the drive, and handle multi-core cable which supplies current to the various motors which actuate the tool boxes and traverses. In almost every case a " Wayne " reel has to be included for remote-control purposes, that is, the attendant is able to control one or more of the motors from a centrally placed push-button panel. The same remark applies in the case of the lathe where remote control of the motors is necessary.

In general the reels are attached to a ceiling, wall or other convenient point by a flange plate, and when the pay-out is not in a dead straight line are of the swivelling type, turning on a pivot with cable guide. Housed at the side or both sides and totally enclosed is the slip-ring gear, consisting of stout ebonite disc carrying the slip rings, varying in number to correspond with the cores of the cables, made of special lead brass. Also the current pick-up plungers are of high-duty copper fitted with a

renewable copper carbon tip, which gives a high-grade conductivity as well as resistance to wear, whilst the spring of the necessary size and strength to give ample margin for the duty, is of high-grade ribbon steel.

The " Standard " type of fitting, suitable for small hand tools, is treated with special enamel and earthed, whilst all the machines are provided if necessary with an extra slip-ring and plunger connected to the earth wire. A stop-gear mechanism can be included so that the cable when in use need not always be in tension.

Further with regard to the " Major " and the " Super " types, these are particularly intended for machine tools, and portable tools, taking respectively cables of 0·300–0·625 in. diameter up to 60 ft. long and 0·625–1·250 in. diameter up to 150 ft. long for large machine tools. Included is a floating plunger type of pick-up gear guaranteed to pass a flash test of 1,000–2,000 volts. Either of the two models also can be of the " dual " type for long travel lathes and other tools, that is, with two independently operated drums side by side so that one can pay out whilst the other can wind up, the smallest size, the " baby," taking 10–12 ft. of ordinary twin flex, being particularly intended for the lighting of tools, as well as benches.

Power House Components, Ltd., Albion Chambers, King Street, Nottingham.

Preperite—A New Rust Remover

TO meet the increasing demand for a good, general purpose and economical rust-removing material, The Pyrene Co., Ltd. have recently introduced a new product under the Trade Mark " Preperite " which, although the removal of rust is its primary function, can also be used for the removal of corrosion products from aluminium and aluminium alloys.

Flexible in its application, this material can either be brushed-on or made up for the treatment of parts by immersion in a tank. An immersion " Preperite " solution possesses the important feature that it does not need to be discarded at regular intervals and replaced by a freshly made up solution. This solution will continue in service indefinitely, provided that it is maintained by replenishment with " Preperite " chemical as necessary. The additions required are determined by means of a very simple chemical test. Another feature of this solution is that it can be used cold or heated to any temperature up to 140° F., the warmed solution having the advantage of speed.

When used for the removal of rust a prior degreasing operation is necessary in most cases. Degreasing can be carried out by any suitable method such as solvent wiping (using turps substitute, white spirit), trichlore-thylene or alkali immersion.

" Preperite " is being used for the preparation of steel scuttles and other steel components for Rolls Royce automobile bodies prior to spray gun bonderizing and painting. The method of application adopted is that of " brushing on." When applied by brushing, it is diluted with tap water to a concentration of two parts of " Preperite " to one part of water. In all normal circumstances, a 5-minute application should be sufficient to remove the rust, after which the metal surface may be rinsed off with either hot or cold water.

An immersion " Preperite " solution is made up to a concentration of one part of " Preperite " to two parts

of water for use either cold or hot. The tanks required to hold the solution, in the case of a permanent installation, should be lined either with lead or rubber, but tanks of suitable glazed earthenware can also be used. A number of important immersion installations are already in successful operation. Following treatment by immersion, work may be rinsed, by hot or cold immersion, by hosing or any other suitable method.

The methods by which " Preperite " is applied to aluminium and its alloys are the same as those used for steel. The concentrations of the solution will also, in most cases, be the same, but in certain cases, greater

dilutions may be possible. The treatment leaves an etch which is beneficial for paint bonding.

Other uses of " Preperite " include that of providing a passivation treatment of sheet steel components prior to storage or " C.K.D." shipment in the plain or unpainted state. When it is used for this purpose, no rinsing operation follows the treatment of the work. When " Preperite " is used for the removal of corrosion products prior to " Parkerizing," " Bonderizing " or " Pyluminizing," rinsing is essential.

This new product is supplied in minimum packings of 7-gallon carboys.

Reviews of Current Literature

METALS REFERENCE BOOK

THIS is a first-class reference book in which the major attention is directed to the fundamentals of metallurgy, and although the application of fundamental data is less conspicuous it is presented in a form that leaves nothing to be desired. The need for a book of this type and character has existed for some years, but its preparation was no mean task ; ability of a high order was needed, together with infinite care and patience. Even these necessary attributes would have been insufficient without enthusiasm. Fortunately Dr. Smithells, his associate editors and contributors have combined to meet the need and have produced a reference book of outstanding merit, which will be invaluable to workers in all metallurgical fields.

The thoroughness of the preparation of this book will be appreciated when it is realised that among introductory tables, which occupy 43 pages, are 25 pages of mathematical formulæ of use to the metallurgist and also included is some essential first-aid information for accidents that might occur in carrying out metallurgical work. Forty pages are given to constants of general nuclear physics, while the remarkable change in metallurgical analysis is emphasised by the amount of data given on spectrographic characteristics of the metals and their application to analytical purposes. X-ray crystallography is dealt with at considerable length, while crystallography and crystal chemistry, which includes the structure of metals, metalloids and their compounds, together, occupy 94 pages, details of structures taking up 21 of these. Considerable data is given arising from the study of geochemistry and geophysics, but, as would be expected, much attention is directed to metallography. In this latter section the general nature of the techniques for the preparation of samples for macroscopic and microscopic examination are assumed to be known and are therefore not dealt with in detail, but more attention is given to metallographic methods, the data given being intended as a guide to the investigation of the more commonly encountered metals and alloys and only the most trustworthy methods are recommended. Immediately following is a section devoted to equilibrium diagrams : the diagrams for 377 binary alloys, 86 ternary alloys and 8 quaternary alloys are given. Data on gas-metal systems ; on diffusion in metals ; general physical properties of

metals ; thermodynamics ; thermal conductivity ; thermoelectric properties and temperature measurement ; electrical properties ; special magnetic properties ; all have their place in this book. And the information on these various subjects is not confined to data ; background information is also given, which leads to a fuller understanding.

Not less valuable is that part of the book which commences with mechanical testing and goes on to deal with the mechanical properties of metals and alloys in some detail in 65 pages ; then follows data on deep-drawing properties ; lubricants (it is noteworthy that solid lubricants are omitted) ; foundry data on crucibles and melting vessels, sands, patterns and the usual casting alloys ; refractory materials (the recently developed vermiculite high-temperature insulating bricks are not mentioned) ; fuels, which includes data on the various qualities of metallurgical coke ; carbon and graphite electrodes ; and considerable theoretical and practical information on controlled atmospheres for heat treatment. Although comprehensive tables of the corrosion resistance of all metals cannot be compiled with much accuracy, because of varying factors involved, tables are given which assist in selecting a short list for detailed consideration for which corrosion tests under particular conditions may be necessary ; guidance on suitable methods of test is given, and information on the assessment of corrosion. Data covering the polarographic method of analysis, including a chart of half-wave potentials, are given. The concluding 37 pages are concerned with the electroplating and metal finishing ; welding ; solders and brazing alloys ; some miscellaneous data ; and, as far as the writer has been able to check, the 15-page index at the end is both adequate and accurate.

This book will be invaluable to metallurgists and will meet a long-felt need. In addition to the editors and contributors, the publishers are to be congratulated on the manner in which they have produced what must have been a very difficult book to compose.

W.A.

THE HISTORY OF ENGINEERING IN IPSWICH

THIS book has been published to commemorate the Jubilee of the Ipswich Engineering Society, which was founded in 1899, and the opportunity has been taken to present a brief history of Ipswich from an engineering point of view. The Society was founded on voluntary effort to meet a desire for advanced information upon scientific and technical subjects, and there is no doubt that its efforts have been and still are appreciated. Starting in a small way with about 30 members there has been a gradual growth in membership to nearly 700.

Considerable enthusiasm has been maintained within the Society since its inception and particularly amongst younger members, indicating that it has been well served by its officers.

It was a happy thought that this jubilee effort should be associated with the development of engineering in the town of Ipswich, particularly in view of the expansion of industries in and around the town, not only in variety but also in volume of output. Such well-known firms as Ransomes, Sims & Jefferies, Ltd. ; E. R. & F. Turner, Ltd., and Bull Motors ; Fisons, Ltd. ; Cocksedge & Co., Ltd. ; Ransomes & Rapier, Ltd. ; Reavall & Co., Ltd. ; The Manganese Bronze & Brass Co., Ltd. ; Crane, Ltd. ; Suffolk Iron Foundry (1920), Ltd. ; Egerton (Ipswich), Ltd. ; and British Steel Piling Co., Ltd. ; are admirably described in some detail. These, together with the history of Ipswich and of its environs, and of the Engineering Society, make the book both interesting and informative and it should appeal to the historian as well as to the engineer.

It is unfortunate that Mr. A. E. Newby, M.B.E., M.I.Mech.E., who had collected so much of the information contained in this book, did not live to see it in its completed form. Mr. Newby was one of the original members who founded the Ipswich Engineering Society and it was fitting that he should have been elected President of the Society in its fiftieth anniversary.

The book contains 135 pages, is well illustrated and excellently produced. It has been designed and printed by W. S. Cowell, Ltd., Butter Market, Ipswich.

COBALT

By Roland S. Young. pp. viii + 181, 41 illustrations. Reinhold Publishing Corporation, New York ; Chapman and Hall, London, 1948. Price, 30s.

THE excellence of the well-known series of American Chemical Society Monographs, of which this is No. 108, is too well known to need emphasis, and readers are assured that every precaution has been taken to make the text authoritative and to fill a known need.

The present monograph is written for two classes of reader : (1) The chemist, or other scientific worker, who has occasion to work with cobalt and wants accurate, up-to-date, and complete information ; (2) the chemist or metallurgist who wishes to browse through this field in the hope of adding to his general knowledge or picking up ideas which may be applicable to his own sphere of work.

The author, who is chief research chemist to a mining corporation in South Africa, is admirably qualified, from his own experience, to undertake a work covering such a wide field as this, and he has presented a remarkably condensed yet not indigestible survey which will enable his readers, either to find directly the information they may require about cobalt, or to track it down. After a brief historical and general introduction (in which, perhaps unreasonably, the reviewer was disappointed not to find rather more about the curious early history of the element) two chapters deal excellently with the occurrence and metallurgy of cobalt. In Chapter IV the chemical properties of cobalt and its compounds are covered. It is too much to expect a completely full survey in the space available, but one or two minor points of criticism might be noted. No mention is made of the anomaly by which cobalt appears before nickel in the periodic classification of the elements,

and indeed, little attempt is made to deal with the systematic comparative chemistry of cobalt as distinct from its detailed chemistry. Again, the statement that there does not appear to be as yet any evidence of atomic disruption of cobalt with alpha particles is both inaccurate and a trifle misleading. Not only are isotopes of copper and manganese reported in the literature as produced in this reaction, but breakdowns have also been observed in bombardment with neutrons and deuterons. Then in dealing with the carbonyl on p. 57, only $Co_2(CO)_8$ is mentioned, whereas in discussion of the structural chemistry of cobalt on p. 63, two carbonyls are listed. However, as already mentioned, these are minor details of criticism.

The next chapter is concerned with the physical and mechanical properties of cobalt, and the remainder of the book is devoted to an admirable range of applied chemistry. The place of cobalt in ferrous and non-ferrous metallurgy, and the powder metallurgy of the element are each accorded a chapter, and electroplating, cobalt in the glass and ceramics industries, the catalytic behaviour of cobalt, and finally, its very important significance for the biologist and biochemist are fully covered.

The last chapter deals very adequately with the analytical chemistry of cobalt, both classical and instrumental methods of analysis being discussed and described. There is a good subject index and an author index.

This is an admirably balanced and attractive monograph. The reviewer can best show his appreciation of it by saying that similar reviews for a number of other elements with which the general worker is not too familiar, and where the literature is equally widely scattered, would be a considerable boon.

C. L. WILSON

THE LIGHT METALS INDUSTRY

By Winifred Lewis, B.Sc., XI + 397 pages, with tables, illustrations, schedules, charts and maps. Dmy 8vo., bound in grey cloth covers, price 21s. net. Published by Temple Press, Ltd., Bowling Green Lane, London, E.C.1.

THIS book does not cover the technical ground of the main operational processes of the light metals industry in any detail, but rather sets out from a different angle to view the industry of light metals from a technico-economic approach, attempting to relate it in perspective to the whole structure of modern industry which has the great metal industries as its base. Miss Lewis has achieved her object admirably and her efforts have resulted in a book which is not only interesting but is highly informative. As would be expected, the major part is devoted to the progress of the aluminium industry; indeed this industry takes up the first 15 chapters, while the magnesium industry and a report on the position of beryllium, respectively, are discussed in the last two chapters ; however, the proportion in relation to peace-time consumption of the products of each section of the light metals industry is probably reasonably accurate. Broadly viewed, a more widespread appreciation of the varied and special utility of aluminium has been one of the most compelling forces in the general market expansion since the end of the war. Demand for aluminium products of nearly all kinds has been coming from an increasing number and variety of consumers. The magnesium industry, on the other hand, although it has surmounted great difficulties in production, has not yet

succe..ed in establishing a consumer industry on such a large scale, whilst the use of beryllium is still in its infan.y.

In the first chapter is given a general account of the history. production, properties and uses of aluminium, in which reference is made to raw materials, availability of power, ore processing, reduction of alumina, cost of production of commercial aluminium ingots, the properties of aluminium and an outline of uses in various industries. These are considered in greater detail in subsequent chapters, thus the second, third, fourth and fifth chapters deal, respectively, with bauxite and other ores and their treatment, water power, the aluminium-producing industry, and the aluminium consumer industry. Chapter six is devoted to statistical information relating to the aluminium industry. Particularly useful is the next chapter which gives in tabular form the chemical composition of the light alloys and heavy alloys of aluminium, alphabetically listed under their proprietary names ; the British standards relating to aluminium alloys ; the mechanical properties of the alloys ; the collated physical and electrical properties of some of the best known alloys ; and corrosion data. General information on the casting alloys and of the forging alloys of aluminium are given in two succeeding chapters. The next few chapters are concerned with the working of aluminium and its alloys, joining, and surface treatment, the latter, in particular, being excellently presented. It is noteworthy that a chapter is devoted to super-purity aluminium, while the concluding chapters on aluminium deal with some of the main post-war applications, and with scrap recovery and remelting.

Of the magnesium industry Miss Lewis finds a difficulty in assessing its progress objectively, since its whole effective life-time has been complicated by its war-time career, which has distorted its growth on the production side and interfered with the evidence regarding its prospects on the consumption side, but her introduction to this chapter is well worth reading as is the complete chapter. A similar plan is adopted in dealing with this industry, the history of the industry being briefly reviewed followed by ores and other natural sources of magnesium, production processes, properties of the metal, cast and wrought products in magnesium alloys, surface treatment, and the uses and consumption of magnesium.

While the main problems of the magnesium industry are on the consumer side, those encountered in the beryllium industry are mainly concerned with production, and in the final chapter, devoted to beryllium, the various aspects are reviewed. The book, which includes a useful appendix listing selected reference books in each field, and an index, is admirably written and is an acquisition to the literature on the light metals industry.

B.S.

DIE THERMOCHEMISCHEN EIGENSCHAFTEN DER METALLOXYDE

By Dr.-Ing Wenere Lange, published by Springer Verlag, Berlin 1949 ; pp. 107, 16 illustrations, Price D Mark 12. (In German).

THE book is the enlarged composition of a dissertation written during the war. It deals with a branch of the theoretical metallurgy which was rather neglected in Germany especially in the application of calculation foundations. The fact that thermodynamics is not only a sport of experts in applied mathematics but a useful working tool for metallurgists is becoming more and more appreciated. The present work deals with the affinity equations of carbon oxides, steam and of all important metal oxides. It gives examples for the application of such equations and presents them in numerous figures and tables.

A comprehensive list of literature on the subject (with 132 references) adds greatly to the value of the book for metallurgists.

F.N.

EQUILIBRIUM DATA FOR TIN ALLOYS

Compiled and produced by L. T. Greenfield, M.Sc., A.I.M. and J. S. Bowden, B.Met., F.I.M. and published by the Tin Research Institute, Fraser Road, Greenford, Middlesex. 60 pp. with 36 diagrams ; price 2s. 6d. post free.

THIS publication is intended as a reference book for metallurgists and scientific workers. It replaces an earlier publication prepared by E. S. Hedges and C. E. Homer and the diagrams are presented in a more convenient form, a single diagram now being given for each system. This has been made possible, in some cases, by combining the results of two investigators. Interpretation of the diagrams is facilitated by the introduction of temperature and composition co-ordinates, and confusion with phase boundaries, solidus, and liquidus curves have been avoided by printing the co-ordinates in colour. Additions have been made to the earlier list of binary alloy systems and all the useful systems, investigated to date, are represented. Supplementary explanatory notes are appended to many of the diagrams and, where available, information on crystal structure is given.

A bibliography of publications on ternary tin alloy systems is included which contains references to all the ternary systems partly or wholly investigated at the time of going to press. Notes are included indicating the extent of the information available where an investigation is incomplete. Owing to their greater complexity, in comparison with binary systems, ternary systems cannot be condensed to a state where they can be adequately represented by one or two diagrams and the bibliography will assist the research worker in locating the original publications to be consulted for these systems.

For the convenience of readers overseas copies of this publication are available at : Tin Research Institute Inc., 492, West Sixth Avenue, Columbus 1, Ohio, U.S.A. (50 cents) ; Centre d'Information de l'Etain, 31, rue du Marais, Brussels, Belgium (20 francs) ; N.V. Billiton Maalschappij (Commerciele Dianot), Louis Couperusplain 19, The Hague, Holland (1.40 florins).

N.C.

Data for Spot Welding Mild Steel

A SLIDE rule has been produced by the British Welding Research Association which gives all essential information needed to carry out spot welding operations with mild steel. For material of a given thickness full data is at once available regarding the diameter of electrode, the minimum electrode pressure, weld current, minimum edge distance and weld time. It is a convenient size and will be invaluable in welding shops. It is available at the nominal price of 1s. 6d. post free from the Association, 29, Park Crescent, London, W.1.

E.E has earlier

Forthcoming Meetings

THE INSTITUTE OF METALS

THE Annual General Meeting will be held on March 29-31st, inclusive. The initial meeting will be held at the Café Royal on the morning of March 29th, when the new President will be inducted. At the luncheon which follows the Institute Medal for 1950 will be presented. On March 30th an all day Symposium on " Metallurgical Aspects of the Hot-Working of Non-Ferrous Metals and Alloys " will be held in the Hall of the Institution of Mechanical Engineers. During the morning of March 31st three papers will be discussed : " The Application of X-ray Methods to the Determination of Phase Boundaries in Metallurgical Equilibrium Diagrams " ; " The Mechanism of Deformation in Metals, with Special Reference to Creep " ; and " Recrystallisation of Single Crystals after Plastic Bending."

IRON AND STEEL ENGINEERS GROUP

The 12th Meeting will be held on March 23rd at 4, Grosvenor Gardens, S.W.1. During the morning three papers will be presented for discussion : " Traffic of Iron and Steel Works—A Method of Traffic Analysis " ; " The Application of Very High Frequency Radio Communication " ; and " Structural Frames for Melting Shops." During the afternoon a paper entitled " A Proposed Method of Specifying Travel Motion Performance for Steelworks Overhead Cranes " will be presented for discussion.

THE INSTITUTE OF FUEL

A meeting will be held on March 28th at 5-30 p.m. at The Institution of Mechanical Engineers at which a paper on " High-Temperature Combustion-Heated Furnaces " will be presented by M. W. Thring, M.A., F.Inst.P.

THE BRITISH CAST IRON RESEARCH ASSOCIATION

A Foundry Conference, organised by the above Association, will be held at Ashorne Hill, near Leamington Spa, from March 30th to April 1st inclusive. An extensive programme is arranged covering Chemical Analysis ; Some Light Castings Problems ; Shrinkage and Porosity and Related Problems ; Non-Destructive Testing.

Iron and Steel Institute Awards

The Bessemer Medal for 1950.—To Mr. James Mitchell, C.B.E. (Honorary Treasurer, Stewarts & Lloyds, Ltd.), in recognition of his distinguished contributions to the technical development of the iron and steel industry.

The Sir Robert Hadfield Medal for 1950.—To Mr. G. D. Elliot (Appleby-Frodingham Steel Company, Scunthorpe), in recognition of his contributions in practice and theory to the development of blast-furnace operation.

A Carnegie Silver Medal for 1949.—To Mr. N. H. Polakowski (University College, Swansea) for his Andrew Carnegie Research Report on " The Compression Test in Relation to Cold Rolling."

The Williams Prize for 1949.—Jointly to Dr. D. F. Marshall and Mr. H. C. White (Park Gate Iron & Steel Co., Ltd., Rotherham), for their Paper on " The Conversion to Oil Firing of the Open-hearth Furnace at Park Gate Works."

The Ablett Prize for 1949.—Jointly to Mr. E. L. Diamond (British Standards Institution ; formerly British Iron and Steel Research Association, London), and Mr. A. M. Frankau (British Iron and Steel Research Association, London), for their Paper on " Present Methods of Open-hearth Furnace Charging."

PROTOLITE, LTD., of Central House, Upper Woburn Place, London, W.C.1 have changed the Telephone No. of their London office to EUSton 8265, and the Telegraphic Address to : Prolite, Kincross, London.

NU-WAY HEATING PLANTS, LTD. present address is : Vines Lane, Droitwich. The Telegraphic Address : Jasnu, Droitwich ; and Telephone No. Droitwich 2331.

W. & J. GEORGE & BECKER, LTD., of Nivoc House, 17–29, Hatton Wall, London, E.C.1, have added three telephone lines to their private branch exchange. The new numbers are HOLborn 5483–5.

Fuel Efficiency Exhibition
Samuel Fox & Co. Ltd.

IN the leading article in this issue, reference is made to the main aspects of the problem of the efficient utilisation of fuel in the iron and steel industry. At the works of Samuel Fox & Co., Ltd., at Stocksbridge it is felt that fuel efficiency is the responsibility of all and not of the Fuel Engineering Department alone. Accordingly, during the last few years a Fuel Efficiency Organisation has been developed with the basic idea of improving the fuel efficiency of the works through the efforts of all employees. So that the whole works could be under constant surveillance, volunteers were called for, who would join the organisation and who would, during their working hours, watch for and report any sign of inefficiency. These reports are made, either through an intermediary such as a foreman or manager, a member of the departmental fuel efficiency committee, or direct to the Fuel Efficiency Organiser who is a member of the Fuel Engineering Department. One in every six employees is a member of the organisation and monthly meetings are held in each department to discuss various problems relating to efficiency. Lectures and the distribution of information of interest to members helps to keep the organisation efficient and many suggestions and ideas are brought forward and put into use.

The highlight of the year is the Fuel Efficiency Exhibition, held this year from February 20th to February 25th. The object of the exhibition, the only one of its kind in the country, was not so much to teach fuel efficiency as to interest people in the subject and to make them more conscious of its importance in the attainment of a greater output of high-class steel.

Although the exhibits were of general interest, the main theme was Steam Generation, Distribution and Utilisation. Many firms, societies and technical colleges helped by contributing exhibits of material and equipment. Models were exhibited of water tube and shell boilers, locomotives, steam hammers, engines and power generators. Another aspect to which attention was drawn was that of lubrication. The company has had a special lubrication section for little over a year, but in that time a saving of 13% has been effected by more efficient lubrication, which in turn has resulted in a saving in the power requirements of the works.

METALLURGICAL DIGEST

Immersion Pre-Treatments in the Electro-Plating of Aluminium

ALTHOUGH aluminium and its alloys have been electro-plated with various metals for specific purposes for a very long time, and the electro-plate coatings produced have, in a large measure, fulfilled the requirements expected, these requirements have become much more stringent and far more numerous in recent years. Corresponding development work has been in progress and industrial processes have been improved considerably, although the perfect plating from the point of view of economics, ease of production and performance, has not yet been realised. Research at the University of Pennsylvania has been intense with a view to perfecting the preliminary metal dip that is generally used as the basis for adherent electro-plated coatings. The Journal of the Electro-Chemical Society of America, May, 1949, 95(5), pages 205–225, reviews the subject and gives details of this research work in a paper by Samuel Heiman entitled "Deposition of Metals on Aluminium by Immersion from Solutions Containing Fluorides." This shows that a new process has been developed for chemical displacement films of zinc, cadmium and tin on aluminium. The immersion solutions employed contain the metal sulphate together with hydrofluoric acid or the fluoride anion. It is claimed that the metals so deposited have good appearance, sound structure and excellent adhesion of the film, and that this adhesion does not depend on any roughening of the aluminium surface. With the zinc deposit the bond between the zinc and the aluminium is better than it is with cadmium or tin, and it is greater than the cohesive strength of the aluminium base material. The process is therefore advocated as the primary metal dip treatment for the plating of other metals on aluminium. The effectiveness of the process is based on the very high solubility of the oxide film on aluminium in solutions containing the fluoride anion in acid environment. It is this oxide film that normally makes it difficult to obtain adherent immersion coatings on aluminium and the high solubility effect

of the fluoride anion has been demonstrated by potential measurements.

The solutions employed contain the metal concerned as sulphate, hydrofluoric acid or a fluoride salt, and in certain cases organic addition agents. The preferred conditions for zinc deposits on aluminium require zinc sulphate at 5 N concentration, hydrofluoric acid at 1 N and an immersion time of 30–60 seconds at a temperature of 25° C. The process is claimed to be at least equal in quality and simplicity to the zincate process which is regarded as the present best commercial method.

For cadmium immersion deposits the preferred conditions use concentrations of cadmium sulphate 0·035 N, hydrofluoric acid 0·3 N and hydrolysed glue 2 grams/litre. The time of immersion is 5–15 seconds at 25° C.

For tin the concentrations are stannous sulphate 1·5 N, hydrofluoric acid 2·0 N, hydrolysed glue 1 gram/litre, goulac 0·2 grams/litre and purified residue acid 1·3 ml./litre. The immersion time is 5–10 seconds at 25°C.

In the case of copper, immersion deposits were difficult, and although some success was encountered with 24 ST (duralumin type) no success was encountered with 2S (pure aluminium).

Heiman reviews the methods employed for plating aluminium during the previous 50 years, viz :—

(a) *Etching Treatment.*—Etching by means of a mixture of nitric and hydrofluoric acid followed by nickel-plating. This is dependent upon the keying action of the nickel deposit in the pits in the etched aluminium surface.

(b) *Electro - chemical Displacement.*— This includes the various metal dips, iron and manganese in particular, applied from hydrochloric acid solutions. The adhesion here was largely due to the mechanical keying of the metal deposited in the pits in the aluminium rather than true adhesion between two metals. The more recent method of depositing zinc from alkaline zincate solutions is more successful in securing adhesion without pitting.

(c) *Anodic Treatment.*—By depositing a relatively thick porous anodic coating the electro-plated metal adheres by virtue of filling the pores of the oxide coating. This is, in effect, mechanical keying.

(d) Special plating solutions have been proposed or used to a limited extent for the direct plating of various metals on to aluminium with only a limited degree of success in special cases.

(e) *Miscellaneous Methods.*—Adherent copper deposits have been obtained by an immersion process using molten cuprous chloride at 450° C. There are, of course, practical difficulties with this, and additionally the aluminium becomes heat-treated by annealing at this high temperature.

The object of the research was to electro-plate or deposit by immersion, metals other than zinc and obtain a deposit equal to or better than that obtained by zinc immersion process. Consideration of means of dissolving the natural oxide film from aluminium led to the use of the fluoride solutions as the most efficient. The original paper gives much valuable detail and the following summarises the final procedure standardised.

(a) *Alkaline Cleaning*
Inhibited alkaline cleaner 45 g./l.
Time .. .. .. 3–5 mins.
Temperature .. .. 80°–90° C.
(b) Cold water rinse.
(c) Acid dip Bath :
Hydrofluoric acid .. .. 0·5 N.
Time .. .. .. 2–3 mins.
Temperature .. .. 25° C.
(d) Cold water rinse.
(e) Film removal Bath :
Nitric acid (sp. gr., 1·42) 70% by volume
Time .. .. .. 10–20 secs.
Temperature .. .. 25° C.
(f) Cold water rinse.
(g) Immersion dip Bath:
For compositions studied, see above.
Time .. .. .. 5–60 secs.
Temperature .. .. 25° C.
Mild agitation.
(h) Cold water rinse.
(i) Copper strike Bath :
Copper cyanide .. .. 20·0 g./l.
Sodium cyanide .. .. 23·0 g./l.
Sodium carbonate .. 30·0 g./l.
" Free " sodium cyanide 0·3 g./l.
Temperature .. .. 25° C.
pH .. .. 10.
Current density .. 20–30 amp./sq. ft. (2·2–3·2 amp./sq.dm.) for 10 secs., then 5 amp./sq. ft. (0·54 amp./sq. dm.) for about 1 min.
(j) Cold water rinse.
(k) Copper plate Bath :
Copper pyrophosphate solution .. .. (44,45).
Current density .. 30 amp./sq. ft. (3·2 amp./sq. dm.).
Time .. .. .. 18 mins.
Thickness copper .. .. 0·0005in. (0·013mm.).
(l) Cold water rinse.

The results claimed from this new type of process are very impressive, and the practical procedure may prove simpler for production practice and control. Production development work is required in order to establish the merits of the process on miscellaneous types of component, varying alloys and varying forms including castings.

Bright Annealing of Nickel Silver on a Production Basis

By John W. Carter

THE stress-relief annealing of nickel silver is a critical operation in the processing of this important commercial alloy, particularly where a bright clean metal surface must be maintained. This commercial alloy, which contains 65% copper, 18% nickel and 17% zinc, is difficult to bright anneal because of its zinc content.

The International Silver Co. have proved that radiant tube, gas-fired controlled atmosphere, continuous-belt furnaces are an effective means of carrying out the annealing process while maintaining the bright clean metal surface essential for silver-plated tableware blanks. These various shaped blanks are punched from strips of nickel silver, and are rolled and stamped to form individual patterns. During these forming processes, cold-working takes place which requires stress relief of the metal by annealing, usually in the temperature range of 593°–816° C. The hardness of the individual blanks varies with the degree of the cold-working to which the particular areas have been subjected, average values being from 80–90 Rockwell B. Annealing reduces the hardness to values from 30–40 Rockwell B.

In the subsequent stamping operations carefully-formed dies are employed. It is imperative that the annealed blanks be absolutely clean and scale-free to insure maximum die life. Pickling has been used to clean the blanks ; however, a perfectly bright anneal is preferable since it eliminates the handling, fumes and maintenance costs of a pickling line. Several atmospheres are used to prevent the oxidation of nickel silver. Of these, dehydrated, burned ammonia

provides a very satisfactory atmosphere. However, it is expensive because of the price of the ammonia. An atmosphere made from the dehydrated, sulphur-free products of the incomplete combustion of a fuel gas, has provided a perfectly bright annealing atmosphere at approximately one-tenth the cost of an ammonia-base atmosphere.

An approximate analysis of the atmosphere generated for the operation of two continuous mesh belt, bright annealing, radiant tube furnaces at International Silver is tabulated below. The gas used for combustion fuel as well as for generating the atmosphere gas was a manufactured gas having a heating value of 540 B th.u./cu. ft.

CO_2	..	..	..	..	..	5·0%
O_2	..	..	..	..	..	0·0%
CO	..	..	..	..	..	9·0%
CH_4	..	..	..	..	..	0·2%
H_2	..	..	..	..	..	10·9%
N_2 et al	..	..	..	..	79·9%

In the hot zone of the furnace, the equilibrium conditions of the atmosphere shift, materially raising the dewpoint. This rise in dewpoint is due primarily to the water gas reaction :

$$CO + H_2O \rightleftharpoons CO_2 + H_2$$
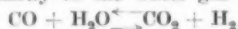
The water vapour thus formed is capable of oxidising zinc either at the work surface or in the form of zinc vapour in the furnace atmosphere.

It is possible by controlling atmosphere flow to flush the hot zone continually with fresh, dehydrated atmosphere. The dewpoint in the hot zone is not too critical to bright annealing at 704°–760° C. In the annealing furnaces at International Silver, the dewpoint is controlled by the rate of atmosphere flow. At the outlet of the hot zone going into the slow cooling zone, the dewpoint must

be maintained at minimum values i zinc oxide is not to be formed on th work. This low dewpoint in the cooling zone is maintained by introducing the major portion of the atmosphere gas at the outlet of the hot zone. The discharge outlet of the furnace cooling zone, the cooling fans' and exhaust hood dampers are set to maintain a positive effluent pressure of atmosphere gas at the charge end of the furnace. This arrangement has the advantage of carrying zinc vapours out the charge end rather than allowing them to contaminate the cooling zone atmosphere and deposit on the annealed work. It also provides a non-oxidising atmosphere for the cooling zone of the furnace. The annealed nickel silver is thus protected and emerges from the furnace bright and clean.

The Determination of Molybdenum and Titanium in Ferro-Alloys and Steels

A method is described by YAKOVLEV and PENKOVA* for the determination of molybdenum in ferro-alloys and steels is based on the precipitation of the iron by sodium hydroxide, followed by reduction by zinc amalgam of the sulphuric acid solution of molybdenum and its titration with permanganate. Methylene blue solution can also be used for the titration, no precipitation of iron being required in this case. The procedure given for the determination of titanium is based on the reduction of this element to the trivalent state by zinc amalgam, followed by titration with ferric chloride in the presence of the thiocyanate of potassium or ammonium. Both the above titrations must be carried out in an atmosphere of carbon dioxide.

* Zavodskaya Laboratoriya, 1949, 15, pp. 34–36. (In Russian).
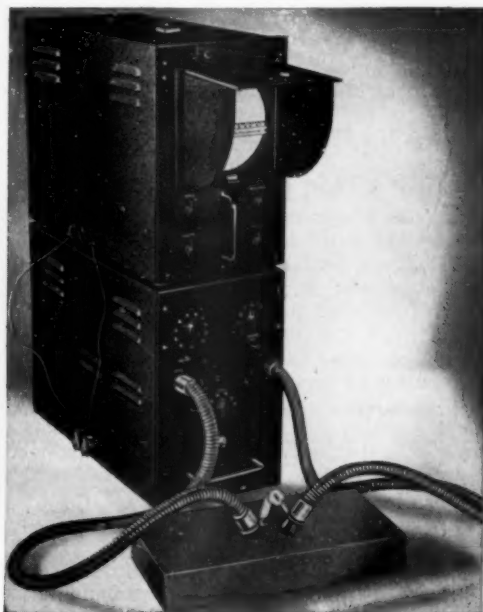
FIND CRACKS, INCLUSIONS, POROSITY
and other defects in welds
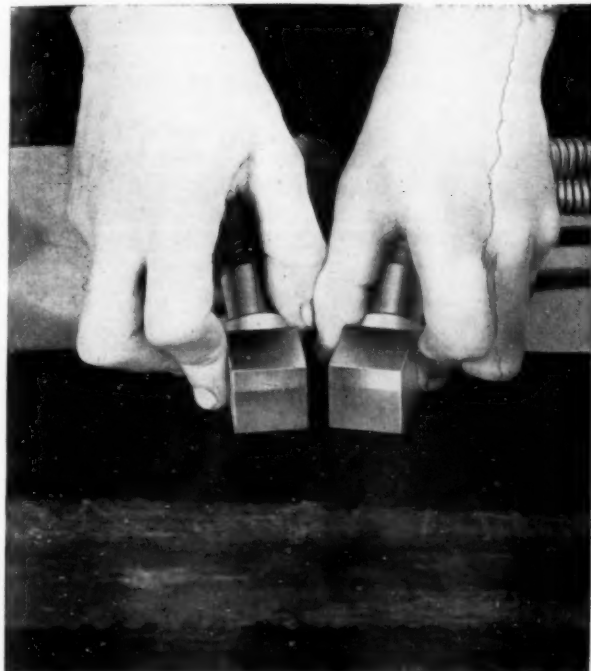
On the left are seen the Transverse Wave Probes in action. Below is shown the Detector. The complete equipment is portable and can be used in practically any situation.

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A Rapid Photometric Determination of Aluminium in Zinc-base Die-casting Alloy Using Solochrome Cyanine R.200

By F. F. Pollak, D.Sc., A.R.I.C. and E. F. Pellowe, B.Sc., A.R.I.C.

A method is described for the rapid determination of aluminium in zinc-base die-casting alloy containing about 4% aluminium. Where the iron and copper contents exceed certain limiting values a preliminary separation is recommended.

SOLOCHROME Cyanine is a water-soluble dye which under specific conditions forms coloured compounds with a number of metal ions—e.g., aluminium, beryllium, magnesium, ferric iron and copper. It has been employed by various workers for the estimation of magnesium in aluminium alloys[1] and of aluminium and beryllium in magnesium alloys.[2,3]

Bacon and Davis[2] have examined the optimum conditions for the photometric estimation of aluminium in magnesium alloys, and from their investigation it would appear that, apart from the dye concentration, the degree of acidity is of paramount importance. The maximum difference between the absorption due to the dye and that due to the aluminium compound has been obtained at 546 mμ with a pH 6·1. However, a slightly higher acidity, pH 5·8, gave a better reproducibility and, in addition, copper and ferric iron interfere least at this pH.

The authors have adapted the method of Bacon and Davis for the determination of aluminium in a zinc-base alloy for die casting (B.S. 1004, Alloy A). This alloy contains only very small amounts of iron and copper and no interference due to these quantities has been observed.

In order to obtain a final pH 5·8, the amount of acid required for dissolving the alloy is critical, and under these conditions solution is very sluggish, even if a few drops of mercuric chloride are added or a platinum wire inserted. Various methods of dissolving the alloy have been tried out and finally a small addition of 100 vols. hydrogen peroxide was found to give most rapid results.

The absorption has been measured with the Spekker Photo-Electric Absorptiometer with Mercury Vapour Lamp, using a filter combination consisting of Chance 6 (OB2) and Wratten No. 62. The manufacture of Wratten No. 62 filters has been discontinued in the meantime, but this filter can be replaced by Wratten No. 74.

For some time past, the use of the left-hand water cell has been considered unnecessary as the absorption due to two reflecting surfaces and a 1 cm. layer of water is

very small. Measurements of the temperature of the photocell with the water cell in position, and without, confirmed that the left-hand water cell can be safely omitted. An adapter has been fitted into the space formerly occupied by the left-hand water cell, which will take additional filters as required. Similar considerations have led to the omission of the right-hand water cell for the setting of the instrument when difference methods are employed. Thus the usual technique[4] has been modified accordingly and will be referred to as " setting air-to-air."

As the excess of dye gives an appreciable blank, a neutral filter (Chance ON32 with density 0·57 at 546 mμ) was added at the left-hand side of the instrument, after setting air-to-air 1·0. By this means nearly the whole range of the drum between zero and 1·0 could be utilised for measurements.

The drum differences have been derived with respect to an aluminium-zinc reference solution because the work of Bacon and Davis, who find that this gives a closer reproduction than a blank, has been confirmed. A 4% aluminium-zinc reference solution has been chosen as this corresponds to approximately the average aluminium content of the alloy to B.S. 1004.

In order to avoid any contamination, all vessels used were of Pyrex glass and were well cleaned with 20% sulphuric acid and then washed free of acid.

METHOD

Solutions Required

(i) *8·21 N. Sulphuric Acid.*

(ii) *Buffer Solution.*—This contains 100 g. sodium acetate ($CH_3COONa.3H_2O$) and 30 ml.N. acetic acid.

(iii) *Dye Solution.*—This is prepared by dissolving 0·25 g. Solochrome Cyanine R.200 in 1 litre of water. It should be allowed to stand for at least 2 hours before use and should be stored in a dark bottle.

(iv) *Stock 4% Aluminium-Zinc Solution.*—0·1 g. aluminium is dissolved in 31·85 ml. 8·21N. sulphuric acid, measured from a burette, with the aid of 5 drops 1% mercuric chloride and 2 ml. 100 vols.

1 Bacon, A., The Absorptiometric Determination of Magnesium. (R.A.E. Report Met. 24).

2 Bacon, A. and Davis, H. C., The Photometric Determination of Aluminium in Magnesium Alloys by Means of Solochrome-Cyanine R.8 (R.A.E. Report No. M.7880A).

3 Wood, C. H. and Isherwood, H., The Use of Solochrome-Cyanine as a Colorimetric Reagent for Beryllium. (*Metallurgia*, 1949, Vol. 39, p. 321).

4 Vaughan, E. J., The Use of the Spekker Photo-Electric Absorptiometer in Metallurgical Analysis.

hydrogen peroxide. After boiling, a solution of 10·55 g. zinc sulphate (ZnSO₄.7H₂O) in approximately 150 ml. water is added and 60·0 ml. 8·21N. sulphuric acid are run in from a burette. The solution is cooled and made up to 500 ml.

(v) 4% *Aluminium Reference Solution.*—An aliquot of 1 ml. of stock 4% aluminium-zine solution (iv) is diluted to 100 ml. This solution should be prepared freshly before use.

Procedure

0·5 g. fine, magnetted drillings of the alloy are dissolved in 20 ml. 8·21 N. sulphuric acid, added from a pipette, with the aid of 2 ml. 100 vols. hydrogen peroxide. When solution has been completed using gentle heat, 20 ml. water are added and the solution is boiled for 5 minutes. After cooling, the solution is made up to 100 ml. An aliquot of 1 ml. is further diluted to 100 ml. and 10 ml. of this diluted solution are transferred to a 100 ml. volumetric flask. An aliquot of 10 ml. 4% aluminium Reference Solution (v) is pipetted into another 100-ml. volumetric flask.

The following additions are made in order :—

50 ml. water.
10 ml. dye solution (iii).

and after mixing :—

5 ml. buffer solution (ii).

The solutions are diluted to 100 ml., mixed and allowed to stand for 10 minutes. Readings are taken using a 2 cm. cell, Chance 6 and Wratten No. 62 (or No. 74) filters, setting air-to-air 1·0 and inserting a neutral filter (density approximately 0·5 at 546 mμ) at the left-hand side after the setting.

The drum difference is converted to percentage aluminium with the help of a calibration graph.

THE CALIBRATION GRAPH
Additional Solutions Required

(vi) *Stock Zinc Solution.*—This is prepared by dissolving 2·2 g. zinc sulphate in 25 ml. water and introducing 18·10 ml. 8·21N. sulphuric acid from a burette.

Next, 2 ml. 100 vols. hydrogen peroxide are added, and the solution boiled, cooled and made up to 100 ml.
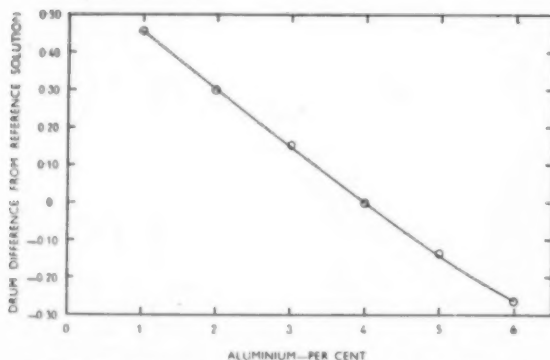
SETTING :—Air-to-air 1·0 with Chance 6 and Wratten No. 62 filters.
Neutral filter (D = 0·57 at 546 mμ) inserted at left-hand side after setting.

Fig. 1.—Calibration graph (1-6% aluminium) for the estimation of aluminium in zinc-base alloy, using the Spekker absorptiometer with a mercury vapour lamp.

(vii) *Dilute Zinc Solution.*—An aliquot of 5 ml. of stock zinc solution (vi) is diluted to 500 ml.

(viii) *Stock Aluminium-Zinc Solution.*—To 1·8 g. zinc sulphate and 0·1 g. aluminium 19·5 ml. 8·21N sulphuric acid are added from a burette and solution is obtained with the aid of 5 drops 1%. mercuric chloride and 2 ml. 100 vols. hydrogen peroxide. After boiling, the solution is cooled and made up to 100 ml.

(ix) *Dilute Aluminium-Zinc Solution.*—An aliquot of 1 ml. of stock aluminium-zine solution (viii) is diluted to 200 ml. This solution should be prepared freshly before use.

Calibration (1-6% Aluminium)

Using burettes of 10 ml. capacity, subdivided into 1/50 ml., the following aliquots are transferred into 100 ml. volumetric flasks.

No.	Dilute Zinc Solution (vii)—ml.	Dilute Aluminium-Zinc Solution (ix)—ml.	% Al
1	9·5	1·0	1
2	9·0	2·0	2
3	8·5	3·0	3
4	8·0	4·0	4
5	7·5	5·0	5
6	7·0	6·0	6

The further procedure is the same as given in the method and the drum differences derived with respect to the reference solution are plotted against aluminium concentration. The calibration graph obtained is shown in Fig. 1.

REPRODUCIBILITY

In order to check the reproducibility of the method, a standard zinc-base alloy which contains 4·03% aluminium has been tested. The following results for the percentage aluminium content have been obtained (each on a separate sample weight).

3·96	3·98	4·10	3·86	4·08
3·98	4·02	4·10	3·93	4·03
3·98	4·04	4·10	4·05	4·10
3·98	4·03	4·09	3·94	4·10
3·98	4·04	4·09	3·99	3·98

Mean = 4·02%. SD = ± 0·066. % SD = ± 1·64.

From this it can be seen that the reproducibility is well within the limits expected for photometric determinations with the Spekker.

INTERFERENCE

Finally, the interference due to iron and copper in amounts exceeding the limits for B.S. 1004 Alloy A has been investigated.

At an aluminium concentration of 4%, iron up to 0·1% and copper up to 0·6% did not give a significant departure from the standard deviation shown above. If, however, both iron and copper were present, the limit was found at 0·075% iron + 0·5% copper and any excess interfered with the method.

As the absorption of iron + copper was different from the sum of the absorption of iron and copper, the construction of a correction graph is rendered difficult.

Where contents of iron + copper above the limit shown above are present, it is recommended that a preliminary separation with a mercury cathode electrolysis cell be employed.

Electrolytic Polishing of Titanium

By D. A. Sutcliffe, J. I. M. Forsyth and J. A. Reynolds*

Royal Aircraft Establishment

Titanium may be satisfactorily polished electrolytically by making it the anode of a cell containing a mixture of acetic and perchloric acids, using a titanium plate cathode and a current density of 30–40 amps./sq. dm. A feature of the method is the short time required to obtain a scratch-free surface.

IN the mechanical polishing and microscopic examination of pure titanium a certain amount of difficulty has been experienced in obtaining the true structure of the material. The false structures obtained have generally been of one of two types. In the first type, a snow-flake structure has been observed on etching, whilst the second type consists of lines similar to those found in cold-worked titanium, which suggests that the

Fig. 1.—Arc-melted titanium—chill cast. × 200

Fig. 2.—Arc-melted titanium—slowly cooled from 1,200° C. × 100

mechanical working of the surface, which has occurred as a result of the polishing operation, has caused the formation of twins. Jaffee and Campbell[1] have observed markings in the grains which they suggest were formed in this way. Both these structures have been obtained by the authors, except when great care has been used in the preparation of the microspecimens.

In view of these difficulties work was started on an investigation to determine the most suitable conditions for the electrolytic polishing of titanium.

The method described has been used successfully with many specimens of various sizes, states of purity and condition.

The electrolyte used was that recommended by Jacquet[2] for chromium—namely, 1,000 ml. of glacial acetic acid and 60 ml. of perchloric acid (sp. gr., 1·54). This bath avoids most of the difficulties normally associated with the perchloric acid technique, in that its explosive tendencies have been decreased and no particular precautions are required whilst mixing the acids. After being in use for several weeks, the electrolyte has shown no signs of deterioration.

The electrolytic cell consisted of a glass tank, containing the electrolyte in which was suspended a cathode made of titanium sheet and having an area greater than that of the specimen to be polished. A suitable distance separating the anode and cathode was found to be between 2 and 4 cm. Current for the cell was taken from the D.C. mains through a large series resistance and the homogeneity of the solution was maintained by a glass stirrer driven by a variable speed motor.

Fig. 3.—Titanium cold worked and annealed 800° C. × 100
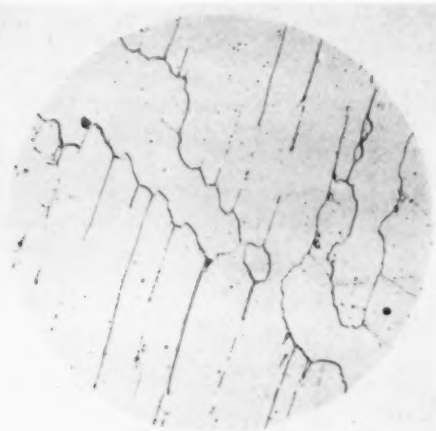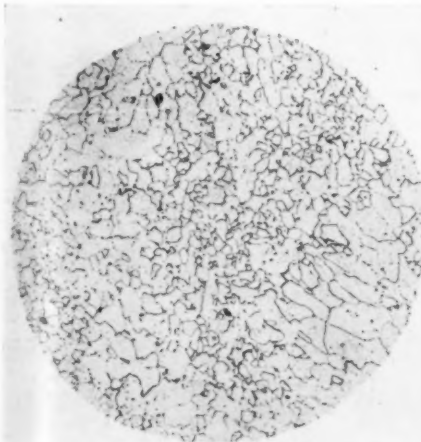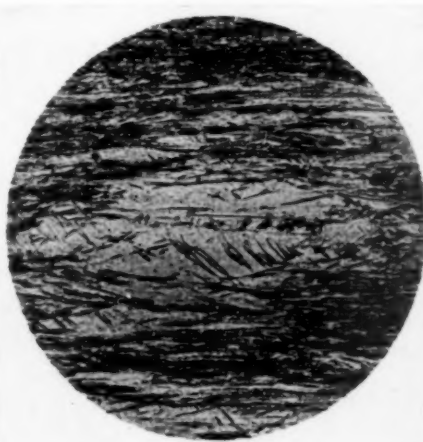

Fig. 4.—Titanium cold swaged 88% × 200
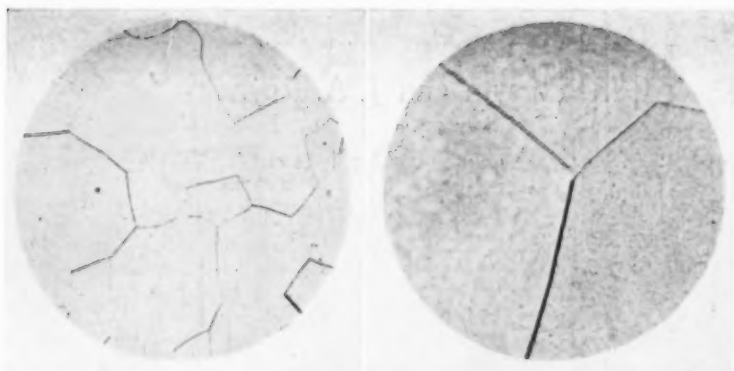
Fig. 5.—Iodide titanium cold worked and annealed. ×100

Fig. 6.—Iodide titanium cold worked and annealed. ×500

on breaking down, also gave rise to pitting of the surface.

Results

The technique described above has been applied to titanium in many conditions and photomicrographs taken on typical examples of specimens prepared in this way and etched in 2·5% nitric acid, 2·5% hydrofluoric acid and 95% water, are shown in Figs. 1–6. The slight loss of focus at the edges of Fig. 5 is due to rounding off at the sides of the specimen which was just over 1 mm. wide.

No evidence of mechanical twinning due to the polishing operation have been found on any of the

Prior to electrolytic polishing, the specimen was prepared by grinding in the usual way to 000 grade emery. It was then suspended vertically in the solution, about 3 cm. from the cathode and the current allowed to flow for about 2 minutes. A current density of 30–40 amps./sq. dm. was found to give the best results in polishing. The time was not too critical, but depended to a certain extent on the preparation of the surface and the purity of the material. Titanium may be left in the bath for considerable periods, as no reaction has been observed when no current is flowing.

When current densities higher than 40 amps./sq. dm. were used, the specimen was found to be severely pitted. Lower current densities than 30 amps./sq. dm. led to the formation of an anodic film on the specimen which,

specimens examined to date, nor have there been any signs of false duplex structures observed.

* Vacation student from Durham University.
1 Jaffe, R. I. and Campbell, J. E., *J. of Metals*, **1**, 9, 646.
2 Jacquet, P. A., *Rev. Met.*, 1949, 46 (4), 214–226.

Polarographic Determination of Lead in Light Alloys

With special reference to Aluminium and Aluminium and Magnesium Alloys with low copper content, and to aluminium alloys with very high copper and iron contents

By W. Stross

International Alloys Ltd.

In the determination of lead by a polarographic method previously described, high results were obtained in the presence of small amounts of copper. Unprecipitated cuprous thiocyanate was found to be responsible for the error and two methods of overcoming the difficulty are described. A difficulty of a different type was encountered on copper and iron hardeners, but this was overcome by slight adjustments of the method.

SOME time ago the author published a method[1] for the polarographic determination of lead in aluminium alloys, which was a simplification of the method of Kolthoff and Matsuyama.[2] More recently he also published his experiences regarding the polarographic determination of lead in magnesium alloys.[3]

The principle of the method is to dissolve the metallics in hydrochloric acid and potassium chlorate, to adjust the pH to approximately 3, and to add hydroxylamine and thiocyanate, thus precipitating copper and reducing iron, whilst stannic tin remains unreduced. The half-wave potentials of the ferrous and stannic ions are much more negative than that of lead and thus do not interfere; the hydroxylamine-thiocyanate treatment reduces the iron but no stannous wave appears after adding hydroxylamine-thiocyanate to the oxidized solution of alloys containing up to, say, 1% of tin.

This method has been used on a considerable scale in this and other laboratories, with good results and with

1 Stross, W., *Metallurgia*, **37** (1947), 49.
2 Kolthoff, I. M. and Matsuyama, G., *Ind. Eng. Chem. (Anal. Ed.)*, **17** (1945), 615.
3 Stross, W., *The Analyst*, **74** (1949), 285.

satisfactory agreement with the spectrographic, gravimetric[4] and dithizone[5] methods.

A. ALLOYS WITH LOW COPPER CONTENT

Poor agreement was, however, occasionally obtained when the method was applied to pure aluminium. When a number of virgin aluminium ingots were tested it was found that some of these consistently gave waves equivalent to 0·015–0·025% of lead, whilst others consistently gave no lead waves or very much smaller ones. In all these cases, however, the dithizone[6] and spectrographic methods, either completely failed to detect any lead at all or, found at the most quantities well below 0·005%.

At this stage of the investigation Dr. W. Cule Davies and Mr. W. Furness very kindly offered to determine the half-wave potential of the wave in question with greater accuracy than is possible with the instrument at the author's disposal. On the two samples of virgin ingots in question, a " positive " and a " negative " one, they confirmed the difference between the two samples but were able to state that what had at first appeared to be a lead wave had a half-wave potential very slightly, but definitely, different from that of lead in the same medium. Their method is as yet unpublished but they kindly authorised the writer to mention that it is a manual procedure, using a Tinsley Vernier Potentiometer, and with calomel reference cells such that the need for correction for I.R. drop is eliminated. The author is greatly indebted to Dr. Cule Davies and Mr. Furness for their co-operation.

The Influence of Copper

It was noticed that the ingots on which these spurious polarographic results were obtained, contained approximately 0·02% of copper, whilst the copper content of the ingots on which no, or only an insignificant quantity of, lead was found polarographically, contained very much less copper.

In their original publication[2] Kolthoff and Matsuyama had stated that, on aluminium alloys containing copper, high lead results were obtained if the final solutions were too acid, as in this case the precipitation of copper is incomplete. The wave of dissolved cuprous thiocyanate coalesces with the lead wave which then becomes too large.

In the author's procedure the pH of the final solutions is even slightly higher than in solutions obtained by Kolthoff's method.[2] The virgin ingots giving the spurious lead results were also tested by Kolthoff's method but the results were, as expected, the same as by the author's procedure.

It seemed, therefore, noteworthy that in all these cases no visible precipitate was obtained after adding hydroxylamine and thiocyanate, only a turbidity or opalescence. The addition of paper pulp (Whatman's accelerator), the use of very close textured filter paper, warming and stirring or prolonged standing of the solutions, singly or in combination, did not induce coagulation, nor did it alter the spurious lead results.

Confirmation of the suspicion that unprecipitated (or incompletely precipitated—see later) cuprous thiocyanate was the cause of the spurious lead results, was obtained in a model experiment.

Super-pure (i.e., practically copper-free) aluminium was treated by the usual technique[1] for lead determination ; the extremely slow attack by the hydrochloric acid was speeded up by adding a quantity of ferric chloride solution equivalent to 0·1–0·2% of Fe. If, in addition, approximately 0·02% of copper were added (as cupric chloride solution) the typical spurious " lead wave " was obtained, whilst no lead wave became apparent when no copper was added. Jewsbury and Osborn[7] have made use of the solubility of very small quantities of cuprous thiocyanate for the determination of the small amounts of copper present as impurity, in high-grade ferrous sulphate, using a thiocyanate medium obviously in order to prevent interference by traces of ferric iron. In evaluating the wave obtained they have to allow for the quantity of lead present (determined in a thiocyanate-free medium) by subtracting from the wave height in thiocyanate medium the equivalent of the lead content. Obviously this procedure could not be applied for the determination of higher copper contents.

After thus establishing the cause of the spurious lead results in these particular virgin aluminium ingots, an attempt was made to prevent the occurrence of a cuprous thiocyanate wave by using hydrazine hydrochloride or hypophosphorous acid[8] in lieu of, or in addition to, the hydroxylamine, at room temperature and at temperatures up to just below boiling point, in the hope that the reduction of the copper would then proceed not only to the cuprous—but even to the metallic state. No improvement was, however, achieved.

It was now considered whether, in alloys of low copper content, the use of thiocyanate could be avoided altogether, as practised, for example, by Nickelson.[9] As the lead wave is sufficiently separated from that of copper in most media—those containing thiocyanate excepted—it should be possible to estimate lead down to, say, 0·01%, in the presence of copper up to, say, 0·03–0·05%, without eliminating the copper, simply by using counter-current. This would cover the practical conditions for, at least, the analysis of pure aluminium. The quantity of iron is, however, often sufficient, even in pure aluminium, let alone in aluminium alloys, to interfere with the lead determination, if it is present in the ferric form. Hydroxylamine alone, without thiocyanate, was found not to reduce the iron completely, as had also been reported elsewhere.[10] A complete reduction of the iron would be achieved during the dissolution of the aluminium in hydrochloric acid but, in this case, without the subsequent use of potassium chlorate, any tin present would also be reduced and would interfere as the wave of the stannous ion coalesces with that of lead.

Successful Methods

The appearance of a cuprous thiocyanate wave can, however, be prevented, either by adding a quantity of copper solution, or by digesting the solution, after the addition of hydroxylamine and thiocyanate, with a few turnings of super-pure aluminium.

ADDITION OF COPPER SOLUTION

If, in the model experiment described above, the quantity of copper is increased sufficiently to produce a distinct precipitate with the thiocyanate, no " spurious lead wave " appears in the polarogram. The addition of $0 \cdot 2\%$ of copper is, in general, satisfactory, but we find it most convenient to add, together with, or before, the hydrochloric acid, sufficient cupric chloride solution to bring the copper content up to the equivalent of approximately $0 \cdot 5\%$ in the metal.

The copper addition in this order of magnitude is without influence upon the true lead wave. If, in the model experiment described above, identical quantities of lead solution (equivalent to, say, $0 \cdot 02 - 0 \cdot 1\%$) are added to two samples, to one of which $0 \cdot 5\%$ of copper is added, whilst no copper is added to the other, identical waves are obtained, thus proving that no co-precipitation of lead with the copper takes place. If, however, only approximately $0 \cdot 02\%$ of copper is added, the lead wave is increased by the " spurious lead wave "—i.e., the cuprous thiocyanate wave.

The fact that the appearance of the cuprous thiocyanate wave is prevented by adding sufficient copper to produce a visible precipitate, makes it seem doubtful whether the appearance of this wave, in the presence of less copper, is a simple function of the solubility of the cuprous thiocyanate ; it seems possible that the effect of adding more copper is the breaking down of a state of supersaturation. It should also be remembered that colloids may also be polarographically active[11] and the effect of adding more copper may, therefore, simply be the flocculation of a colloidal but polarographically active cuprous thiocyanate.

ADDITION OF SUPER-PURE ALUMINIUM

After carrying through the usual procedure[1] up to the addition of thiocyanate and hydroxylamine, add to the solution approximately 50 mg. of fine millings or turnings of super-pure aluminium and digest the solution for about a half hour, with occasional stirring (the temperature being maintained at approximately $70°$ C. by immersion in a water bath), cool, add gelatine, make up to volume, mix and filter off without delay an aliquot through a close texture filter paper (e.g., Whatman No. 542). As the quantity of super-pure aluminium and the time of digestion required are likely to depend upon the fineness of the millings, it seems advisable to check the procedure with every fresh batch of millings. Again it was necessary to check whether losses of lead would occur together with the copper. This was not found to be the case, thus confirming the experience of Kraus and Novak.[12]

Whether with the use of super-pure aluminium the addition of thiocyanate could be dispensed with was not tried out ; it no longer interferes and it is advisable to use it for the sake of uniformity of procedure.

The quantity of superpure aluminium actually dissolved at the low acidity involved is very small, and the risk of also reducing any tin present does not seem to be great but, because of this possibility, and also for greater rapidity, we give preference in practice to the addition of a copper solution.

Apart from the case of the commercial purity aluminium, on which the source of error described above

11 Schrager, B., *Collection of Czech. Chem. Communications*, **1** (1929), 275.
12 Kraus, R. and Novak, J. V. A., *Collect. Czech. Chem. Commun.*, **10** (1938), 554, quoted from Heyrovsky, J., " Polarographie," Springer-Vienna, 1941, 324.

was first observed, a number of commercially important aluminium alloys (e.g., 2L33, D.T.D.300) and practically all the commercial magnesium alloys fall within the same category. Experiments seem to show that other factors than the ratio of copper, lead and iron determine the extent of the copper interference. In view of the danger of interference it seems advisable regularly to use the first procedure described whenever the copper content is below $0 \cdot 5\%$.

B. COPPER AND IRON HARDENERS (MASTER ALLOYS)

It was found that on aluminium alloys containing, say, 50% of copper, or 10% of copper plus 10% of iron, no results could be obtained by the method as described.[1] The reasons for this are : (a) That the prescribed quantities of hydroxylamine and thiocyanate are insufficient ; and (b) that the pH required was not achieved, in consequence of the difference between the equivalent weights of copper (and iron) and aluminium.

The following modifications were found to overcome these difficulties.

(1) COPPER HARDENERS, WITH APPROXIMATELY 50% COPPER

2 N. sodium carbonate solution is used, otherwise the solutions originally described.[1] Use $0 \cdot 6$ ml. of potassium chlorate solution, $6 \cdot 5$ ml. of 2 N. sodium carbonate, 1 ml. each of the hydroxylamine and thiocyanate solutions, otherwise proceed exactly as described.

(2) IRON HARDENERS AND CERTAIN DROSSES

For alloys, containing say 10% each of copper and iron (e.g., drosses) the procedure is similar. Use $0 \cdot 3 - 0 \cdot 6$ ml. of potassium chlorate solution, as necessary. Use 11 ml. of 1 N. sodium carbonate solution and 1 ml. each of the hydroxylamine and thiocyanate solutions. Even these increased quantities may not completely reduce the iron in the case of high iron contents (as shown by the persistance of the ferric thiocyanate colour) but this is achieved by immersing in water of about $80°$ C. for a few minutes.

For copper and iron contents between those defined in the preceding paragraphs intermediate quantities of sodium carbonate have to be used, to achieve the desired pH of $3 \cdot 0 - 3 \cdot 5$, which can be checked with narrow-range indicator papers. It might appear simpler to use in such cases the original method of Kolthoff and Matsuyama,[2] in which thymol blue is added and the reaction of each sample adjusted individually ; the strong colour of the large quantities of iron and copper involved makes this, however, impracticable.

For hardeners and other samples of unusual composition the method of internal standards—i.e., the addition of suitable quantities of standard lead solution to a duplicate sample, is recommended, as the calibration data established on typical alloys cannot be applied directly.

MR. JAMES M. MOWAT, Chief of Research at the Parkhead Steel Works of William Beardmore's, has been appointed a Special Director of the company.

Primary Analytical Standards

By A. J. Nutten

Department of Chemistry, The University, Birmingham

Continuing the consideration of the various substances which have been advanced as primary analytical standards, the author concludes with a series of substances applicable to oxidimetry and argentometry.

II. OXIDIMETRIC

Potassium Dichromate

$K_2Cr_2O_7$. Eq. Wt. 49·04.

Indicators : o-Phenanthroline/Starch/Diphenylamine Type.

Potassium dichromate is perhaps the most widely used oxidimetric standard. It conforms to all six requirements of a primary standard, its colour not interfering with the end-point colour of the redox indicators used. As a titrant for dilute ferrous ion, potassium dichromate possesses several advantages over permanganate. Thus it is easily obtained in the purest state and is absolutely permanent in acid solution. Its oxidations take place immediately and in the cold, in the presence of free hydrochloric or sulphuric acids.

Jander and Beste[81] found that a 0·1 N solution of potassium dichromate (15 ml.) when added to a cold solution of 2 N potassium iodide (40 ml.) and concentrated hydrochloric acid (40 ml.) will liberate an equivalent weight of iodine within 15 minutes. Values obtained by these workers were in close agreement with those obtained by other methods. The iodine solution must be titrated fairly slowly, otherwise high results will be obtained.

Vosburgh[82] describes experiments which indicate that the reduction of potassium dichromate by iodide is quantitative if the solution contains 2–3 g. potassium iodide for 5 milli-equivalents or less of dichromate, and if the reaction is allowed to proceed in the dark for 4–10 minutes. The solution is then diluted, and titrated with sodium thiosulphate.

From the numerous papers appearing in the literature, there is no doubt that, if all known sources of error are avoided, potassium dichromate is an excellent primary standard.

Hexanitrato Ammonium Cerate

$(NH_4)_2 Ce (NO_3)_6$. Eq. Wt. 548·26.

Indicator : o-Phenanthroline.

Smith, Sullivan and Frank[83] describe the preparation of hexanitrato ammonium cerate, and state that, when so prepared, it is an oxidimetric standard of the first order.

The starting material was ceric oxide (free from thorium, iron and manganese) containing approximately 46% cerium oxide. The concentrated nitric acid solution of the ceric oxide is evaluated in terms of the $H_2Ce (NO_3)_6$ content, and an excess of the theoretical amount of crystalline ammonium nitrate added to the hot solution. Orange crystals of hexanitrato ammonium cerate deposit. These are filtered off, dissolved in water, and the solution concentrated to crystallisation. The first two crops of crystals are combined, and dissolved in concentrated nitric acid (270 ml. acid per 100 g. crystals). A slight excess of ammonium nitrate is added, and the pure hexanitrato ammonium cerate

obtained on cooling is filtered off and dried at 110° for 1½ hours. Recrystallisation from concentrated nitric acid in the presence of 15–30% excess ammonium nitrate gives a product, the composition of which was found to be theoretical within ordinary analytical accuracy.

The above workers state that solutions of the nitrato-cerate in 0·5 N–2 N sulphuric acid are perfectly stable at 100° C. and that it is by far the most important reagent for use in preparing solutions as standards for cerate oxidimetry.

Smith and Getz[84] used hexanitrato ammonium cerate dissolved in 1 N or 2 N nitric acid or perchloric acid for direct titration of oxalate, the high oxidation potential of these reagents causing an increased rate of oxidation.

Potassium Bromate

$KBrO_3$. Eq. Wt. 27·84.

Indicator: a-Naphthoflavone/Starch.

Potassium bromate in hot acidified solution reacts with certain reducing agents, forming potassium bromide. When the reducing agents have been completely oxidised, a further drop of bromate solution will react with the bromide, liberating free bromine which may be detected by its decolorisation effect on methyl orange and other azo dyes, or, better, by the colour produced by its reaction with a-naphthoflavone indicator.

Yakowitz[85] states that pure potassium bromate is easily prepared by three recrystallisations from hot water, and drying of the product at 140° C. It is very stable, and standard solutions can be prepared by weighing the pure salt to give a solution with a strength known within 0·05%.

Kolthoff[86] confirms that potassium bromate is easily prepared in a pure state and that it is stable.

A sample of the salt was kept by Yakowitz for 16 months, under laboratory conditions, in a glass-stoppered amber-coloured bottle. During this time, solutions of the bromate were prepared containing known weights of salt in known volumes of solution. These solutions were checked against standard arsenious oxide and sodium thiosulphate solutions. In each case the calculated normality agreed closely with the experimental value.

Other workers have fully substantiated the claim that potassium bromate is a suitable primary standard for oxidimetry.

Potassium Iodate

KIO_3. Eq. Wt. 35·67.

Indicator : Starch.

Potassium iodate can readily be procured in a pure state by recrystallisation from hot water. It can be weighed directly, being used to standardise thiosulphate solutions. It reacts quantitatively with an acidified solution of potassium iodide according to the equation :

II. OXIDIMETRIC

81 Jander, G. and Beste, H., *Z. anorg. allgem. Chem.*, **133**, 73 (1924).
82 Vosburgh, W. C., *J. Am. Chem. Soc.*, **44**, 2120 (1922).
83 Smith, G. F., Sullivan, V. R. and Frank, G., *Ind. Eng. Chem. (Anal. Ed.)*, 449 (1936).

84 Smith, G. F. and Getz, C. A., *Ind. Eng. Chem. (Anal. Ed.)*, **10**, 304 (1938).
85 Yakowitz, M. L., *J. Assoc. Official Agr. Chem.*, **18**, 505 (1935).
86 Kolthoff, I. M., " Volumetric Analysis," p. 367, J. Wiley & Son, New York (1939).

$$KIO_3 + 5KI + 6HCl \longrightarrow 6KCl + 3I_2 + 3H_2O$$

The stability of potassium iodate solutions has been questioned by Sinton,[87] although Jamieson[88] finds that its solutions are permanently stable.

Berman[89, 90] standardised potassium iodate by means of analytically pure arsenious oxide as follows :—

0·1 g. arsenious oxide are weighed into a 250-ml. glass-stoppered flask, 30-ml. concentrated hydrochloric acid added and solution effected. Five ml. chloroform are then added, and the solution titrated with M/40 potassium iodate solution, rapidly at first till the liberated iodine is largely gone, and then slowly. At the end-point, the chloroform loses the last trace of pink due to the free iodine and is colourless, the aqueous solution retaining a pale-yellow colour. If more than 25-ml. iodate solution is used in the titration, 10–15-ml. concentrated hydrochloric acid is added to maintain the necessary acidity. The results obtained by Berman were consistent over a two-year period, the iodate solutions retaining their original titre value.

It may be concluded that potassium iodate can be sufficiently purified, and is sufficiently stable in the air or in solution to be used as a standard substance.

Potassium Biiodate

KH(IO₃)₂. Eq. Wt. 32·5.
Indicator : Starch.

Potassium biiodate has already been described as an alkalimetric standard. It is also widely used in iodometry, its one disadvantage in this sphere being its low equivalent weight. The method of preparation by Shaffer and Hartmann[10] may be outlined :—

110 g. potassium chlorate are added to 450-ml. warm water containing 40-ml. concentrated hydrochloric acid in a 2-litre Erlenmeyer flask, and placed in a well-ventilated hood ; 100 g. powdered iodine are added and the mixture warmed with occasional shaking till the reaction starts. When the reaction has subsided, the solution is boiled for a few minutes and filtered hot by suction. On cooling, the solution deposits about 150 g. biiodate (about 90% pure) which is then filtered by suction, redissolved in about three times its weight of boiling water and filtered hot. After standing overnight, the crystals are filtered by suction and dried at 120°.

Shaffer and Hartmann consider one recrystallisation sufficient to obtain pure biiodate, but most other workers recommend the second or third recrystallisation for complete purity.

Cupric Oxide

CuO. Eq. Wt. 79·57.
Indicator : Starch.

Electrolytic copper has been used industrially for the standardisation of thiosulphate solutions. Buehrer and Mason[91] have shown that the assumption that electrolytic copper is 100% pure is incorrect. Their experiments have led them to propose cupric oxide as a suitable standard particularly because of its uniformity of composition, and they have devised a method of preparation to prevent occlusion and adsorption during the precipitation, and to ensure complete dehydration on ignition without reduction to the metallic form.

Cupric oxide was prepared in four different ways, and its purity determined electrolytically. Values were

obtained which were less than the theoretical percentage of copper, but uniformity of composition was indicated. On the basis of their findings, Buehrer and Mason consider the oxide trustworthy as a standard. It seems justifiable, however, to comment on the use of a purity factor for each batch of oxide, a procedure which precludes the use of cupric oxide as a primary oxidimetric standard.

Arsenious Oxide

As₂O₃. Eq. Wt. 49·46.
Indicator : o-Phenanthroline/Starch.

It is well known that direct titration of trivalent arsenic with permanganate in dilute acid solutions is unsatisfactory since the reaction does not proceed stoichiometrically. Lang[92] states that a minute amount of iodide or iodate acts catalytically to complete the reduction of manganese to the divalent state.

Kolthoff, Laitinen and Lingane[93] compared the normality of 0·5 N permanganate determined by Lang's procedure with that found by direct potentiometric titration of potassium iodide with permanganate in very dilute acid solution. The factors obtained by the two methods agreed to within 0·03%.

Bright,[94] using Lang's catalytic method, standardised 0·1 N permanganate solutions against Bureau of Standards arsenious oxide. His normalities were compared with those obtained by Fowler and Bright[95] (who used pure sodium oxalate for the permanganate standardisation) and were found to agree to within 1 part in 3,000.

According to Willard and Young[96] standardisations of potassium dichromate against arsenious oxide can be made accurately using osmium tetroxide as catalyst and o-phenanthroline-ferrous complex as indicator. At 50° C. a slight excess of dichromate is required, indicating that the reaction is too slow for a visual end-point. Using a potentiometric method at still higher temperatures, these workers obtained good results, due to an increase in reaction velocity. The procedure, however, is lengthy and is not recommended.

The uses of arsenious oxide are many and are listed in most textbooks on quantitative analysis. There is no doubt that it is a most excellent and direct primary standard substance.

Sodium Oxalate

Na₂C₂O₄. Eq. Wt. 134·01.

Sodium oxalate is readily obtained in a pure state and may be dried by heating for 2 hours at 120° C. It is non-hygroscopic.

The work of Hopkins[97] indicates that sodium oxalate solutions for use in water analysis deteriorate considerably after one week, and after seventeen weeks only 74% of the theoretical amount of permanganate is required. If, however, 100-ml. dilute sulphuric acid (1 : 4) is present per litre of sodium oxalate solution, no deterioration is detected after two months, even when in clear glass bottles in daylight.

The usual procedure of McBride[98] for the standardisation of permanganate by sodium oxalate is stated by

87 Sinton, F. C., *J. Assoc. Official Agr. Chem.*, **15**, 420 (1932).
88 Jamieson, G. S., A.C.S. Monographs, Chem. Catalogue Co.
89 Berman, S. M., *J. Assoc. Official Agr. Chem.*, **19**, 537 (1936).
90 Berman, S. M., *J. Assoc. Official Agr. Chem.*, **20**, 590 (1937).
91 Buehrer, T. F. and Mason, C. M., *Ind. Eng. Chem. (Anal. Ed.)*, **1**, 68 (1929).

92 Lang, R., *Z. anal. Chem.*, **152**, 197 (1926).
93 Kolthoff, I. M., Laitinen, H. A. and Lingane, J. J., *J. Am. Chem. Soc.*, **59**, 429 (1937).
94 Bright, H. A., *Ind. Eng. Chem. (Anal. Ed.)*, **9**, 577 (1937).
95 Fowler, R. M. and Bright, H. A., *J. Research Natl. Bur. Standards*, **15**, 493 (1935).
96 Willard, H. H. and Young, P., *Ind. Eng. Chem. (Anal. Ed.)*, **7**, 57 (1935).
97 Hopkins, E. S., *Ind. Eng. Chem.*, **15**, 149 (1923).
98 McBride, R. S., *J. Am. Chem. Soc.*, **34**, 393 (1912).

For[?]r and Bright[95] to give high results when compared with values obtained by using other primary standards, the error being as high as 0·4%. In their recommended procedure, 90–95% of the permanganate is added to a dilute sulphuric acid (1 : 19) solution of sodium oxalate at 25°–30° C., the solution warmed to 55°–60° C. and the titration completed.

Sodium oxalate has found widespread use as a primary standard in oxidimetry.

Oxalic Acid

(COOH)₂.2H₂O.   Eq. Wt. 126·07.

Potassium Tetroxalate

KHC₂O₄.H₂C₂O₄.2H₂O.   Eq. Wt. 127·1.

These two substances, especially oxalic acid, have been widely used in routine analytical work, but are not to be recommended as standard oxidimetric materials due to the uncertainty as to their true water contents.

Ferrous Ethylenediamine Sulphate

(CH₂.NH₂)₂ H₂SO₄.FeSO₄.4H₂O.   Eq. Wt. 382·14.
Indicator : o-Phenanthroline.

Ferrous ethylenediamine sulphate (FES) first prepared by Grossman and Schück[99] was suggested by Oesper and Caraway[100] as a primary oxidimetric standard.

FES is prepared as follows :   60-ml. 6 N sulphuric acid are added to 10 g. 98% ethylenediamine solution : 46·3 g. pure ferrous sulphate are dissolved in the acidified solution, water added to 300 ml. and then 300 ml. ethanol. The precipitate is filtered, washed with 50% ethanol, and redissolved in slightly acidulated water. Ethanol is then added to give a 3 : 2 solution ethanol ratio. The precipitate is filtered, washed with 65% and 95% ethanol, and dried in an oven overnight at 50° C.

When so prepared FES is a fine-grained, very pale-green, crystalline compound. Data given by Oesper and Caraway indicate that it reacts stoichiometrically with standard solutions of ceric sulphate, potassium permanganate and potassium dichromate.

The salt is stated to be stable indefinitely under ordinary laboratory conditions, the water of hydration not impairing the accuracy of the results obtained. The high equivalent weight is a distinct advantage.

The homologous propylenediamine compound has been prepared[101] and has also been found acceptable as a primary oxidimetric standard.

A direct standard for use in cerate oxidimetry has long been lacking. The two compounds above provide this missing link and should open the way to further research on the preparation and properties of other ferrous organo-inorganic compounds for use in oxidimetry.

III. ARGENTOMETRIC

Sodium Chloride

NaCl.   Eq. Wt. 58·45.
Indicator : Dichlorofluorescein.

The A.R. compound is sufficiently pure for use as a primary argentometric standard. Commercial material may be purified by passing hydrogen chloride into a saturated solution of the non-iodised salt. The precipitate is filtered, washed several times with water, and dried. If purity tests are not satisfactory, the salt is then heated to constant weight in a crucible over an alcohol flame, or in an electric oven at a temperature of 500°–600° C.

Purity tests for sodium chloride have been listed by Kolthoff and Stenger.[102]

Potassium Chloride

KCl.   Eq. Wt. 74·55.
Indicator : Dichlorofluorescein.

The A.R. product is generally sufficiently pure for argentometric standardisations not of the most precise nature. The salt may be purified by three recrystallisations from distilled water, followed by similar heat treatment as described under sodium chloride.

Potassium Thiocyanate

KCNS.   Eq. Wt. 97·17.

The use of pure potassium thiocyanate as a primary standard substance has been discouraged in the literature. Kolthoff and Lingane,[103] however, made a thorough investigation into the preparation of the pure salt, and concluded that it is a suitable standard for work requiring an accuracy not greater than 0·1%.

Samples of potassium thiocyanate were repeatedly recrystallised from water, ethanol and methanol. Other samples of the pure salt were prepared by fractional precipitation from a saturated ethanol solution by the addition of ether. The crystals were heated to 200° C. and kept melted (M.P. 172° C.) from 10–20 minutes to remove the last traces of solvent.

The salt showed no increase in weight after standing for a week over deliquescent calcium chloride hexahydrate (relative humidity 0·3) and for a week longer over potassium carbonate dihydrate (r.h. 0·45). Increase occurred over deliquescent sodium bromide dihydrate (r.h. 0·57).

The experimental findings of Kolthoff and Lingane showed that the end-point of a potentiometric titration of silver with thiocyanate can be found with an accuracy of less than 0·01%.

The Volhard method of determining the end-point leads to an effective strength 0·1% greater than the potentiometric titration, due to occlusion of silver nitrate at the end-point. If the precipitate is digested just before the end-point, the Volhard method gives results almost identical with the potentiometric method.

Silver Nitrate

AgNO₃.   Eq. Wt. 169·89.
Indicators : Dichlorofluorescein/Eosin.

Silver nitrate is obtainable commercially in a highly purified state. If it contains traces of impurities, it may be purified by recrystallisation from water containing a small amount of nitric acid, followed by filtration on a sintered glass crucible.

An anhydrous product is obtained on melting the salt and keeping it in this condition (c.a. 210° C.) for 5 minutes. Rosa, Vinal and McDaniel[104] state that slight decomposition results if the molten compound is kept at more elevated temperatures.

Silver

Ag.   Eq. Wt. 107·88.

Though metallic silver of very high purity may be prepared electrolytically, the most convenient method,

95 [?]ossman, H. and Schück, B., *Z. anorg. Chem.*, **50**, 29 (1906).
100 [?]sper, R. E. and Caraway, K. P., *J. Chem. Educ.*, **24**, 235 (1947).
101 [?]tten, A. J. *Anal. Chim. Acta*, **3**, 433 (1949).

102 Kolthoff, I. M. and Stenger, V. A., "Volumetric Analysis, Vol. 11," p. 251. Interscience Publishers, New York (1947).
103 Kolthoff, I. M. and Lingane, J. J., *J. Am. Chem. Soc.*, **57**, 2126 (1935).
104 Rosa, E. B., Vinal, G. W. and McDaniel, A. S., *Bull. Bur. Standards*, **9**, 524 (1913).

and one yielding an equally pure product, is that of Richards and Wells.[105] These workers reduced silver nitrate with ammonium formate :—

$$2 \ HCOONH_4 + 2AgNO_3 \longrightarrow 2Ag + CO_2 + 2NH_4NO_3 + HCOOH$$

A solution of ammonium formate, prepared by passing

105 Richards, T. W. and Wells, R. C., *J. Am. Chem. Soc.*, **27**, 459 (1905).

ammonia gas into freshly distilled formic acid, is trea ed with pure silver nitrate. The precipitated silver is filtered and washed with distilled water : it is then fu ed in a stream of hydrogen in a calcium oxide boat.

The product conforms to the specifications o: a primary standard substance, but is not as conveni nt as the more common silver nitrate.

Miscellaneous Microchemical Devices (XXIV)
Holding and Clamping Devices
By J. T. Stock and M. A. Fill

A FEATURE possessed by all of these devices is the utilisation of ordinary bakelite reagent-bottle caps.

The base of the stand shown in Fig. 1 is a cap A of some 60 mm. outside diameter. A hole is drilled centrally in it and a 50-mm. length of 8-mm. bore heavy-walled glass tubing B is fixed in by sealing-wax. The upper end of the tubing is lightly flared. Jaws CC are bent from 3-mm. diameter glass rod as shown. After the vertical portions have been passed through B, they are again bent at right angles and approximately parallel to the jaws as shown in Fig. 2, which is a view of the underside of the base. Gripping force is provided by a rubber band D cut from thin-walled tubing and sprung across the ends of the rods, as shown.
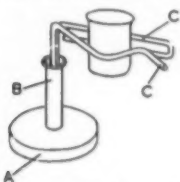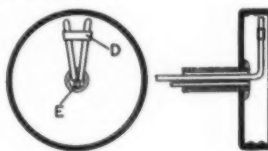

Fig. 1.—Bench-type clamp.

Fig. 2.—Details of construction of clamp.

If one jaw is placed between the first and second fingers and pressure applied to the other jaw by the thumb, the device may be operated by one hand. Cylindrical objects, such as test tubes in various sizes, drop-pipettes, etc., may then be clamped or withdrawn as required. To prevent wedging action, it is desirable to insert in tubing B a piece of glass rod E. This should have a length about that of B and should be arranged behind the vertical portions of the jaws, as shown in Fig. 2.

The tongs shown at (a) in Fig. 3 are useful for recovering microscope slides, crucibles, etc., from a chromic acid bath ; the ends of the jaws F and G may obviously be modified as at (b) for handling cylindrical objects. Quite a wide opening is obtainable, so that small beakers may be accommodated. Two 35-mm. outside diameter bakelite bottle caps HH, retained mouth-to-mouth by a No. 8 B.A. brass bolt J and nut, provide for the retention of the jaws. Except for the bolt-hole, one cap requires no alteration. Gaps in the walls of the other are cut to a depth a little greater than the diameter of the glass rod used to make the jaws as shown unshaded in C, Fig. 3. To locate the jaws in position, small spheres are formed upon them as shown at (d), the distance apart being a few mm. greater than the diameter of the caps. This view also shows how a band of rubber K cut from thin-walled tubing provides the closing force of the tongs. Before assembly, a small pad L, cut from a

rubber bung, is placed beneath fixed jaw F. When the bolt is tightened, F is then securely gripped by being pressed upon this pad.
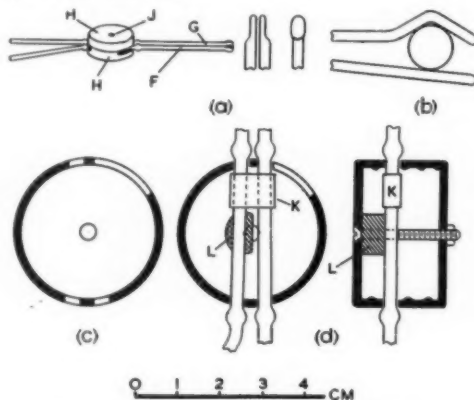

Fig. 3.—Tongs for corrosive conditions.

Friction drive is utilised to raise and lower the boss-head M of the stand shown in Fig. 4. Light attachments such as the glass clamp previously described,[1] a Belcher

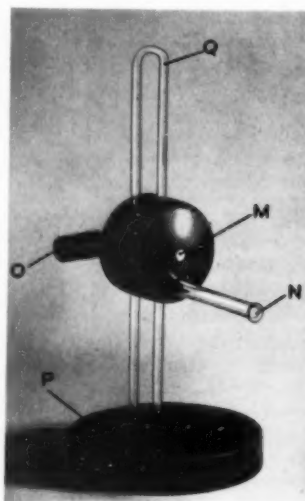1 Stock, J. T., and Fill, M. A., *Metallurgia*, 1947, **35**, 172.

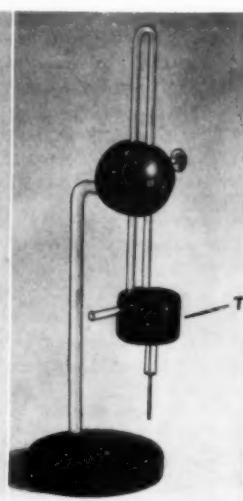Fig. 4.—Friction-drive boss-head and stand.

Fig. 6.—Micro-stirrer with lowering device.

microspoon[2] or a glass-rod frame for holding microscope slides are inserted into tube N projecting from the face of the boss-head, and are retained by a sleeve of thin-walled rubber tubing. Operation is by rotation of knob O.

The base P of the stand may be a wood block or a large bottle-cap. In the latter case, the under side is filled with a mixture of sealing-wax and lead shot. "Hairpin" Q, is bent from 3-mm. diameter glass rod, so that the gap is about 6 mm. Care should be taken to get the arms as nearly parallel as possible.

Bosshead M consists of two 35-mm. diameter bottle caps bolted mouth-to-mouth. The front cap carries tube N, the rear one the operating mechanism. Details of the latter are shown at (a) and (b) in Fig. 5. Two pairs of holes to allow passage of the arms of the "hairpin" are drilled parallel and close to the under side of the rear cap. About 5 mm. above centre (to avoid fouling the clamping bolt) and about 3 mm. from the mouth, a third pair of holes is drilled to accommodate operating spindle R. This pair of holes should be at right angles to the other two pairs. The operating spindle is merely a short length of 3-mm. diameter glass rod. Knob O is either formed by thickening up the
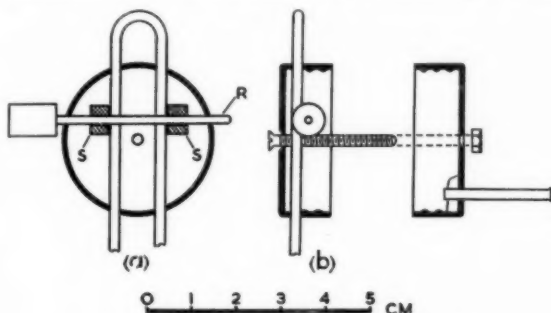

Fig. 5.—Mechanism of friction drive.

glass or is of bakelite and cemented on. Driving rollers SS are about 10 mm. in diameter and are cut from appropriate-sized borings of a rubber bung. The pointed tang of a file or other sharp tool is then thrust axially through the centre of each.

To assemble, the arms of the "hairpin" are slipped through the holes in the rear cap and cemented in the base. The spindle is then inserted through one of its holes, the driving rollers forced on, and the spindle fed through the second hole after adjusting the positions of the rollers. The inside faces of the latter should grip the outsides of the arms, so that, on turning the knob, the cap moves along the arms. When the adjustment is satisfactory, the motion should be smooth but the cap should show no tendency to move downwards when a load of about 200 g. is applied to it. Bolting-on of the front cap completes the assembly.

A modification that is sometimes useful is to keep the bosshead fixed while the "hairpin" moves. An example is the stirring device shown in Fig. 6. The stirrer unit T may be a small vacuum-operated one[3] having a body made of bottle-caps. Alternatively, these bottle-caps may enclose a miniature electric motor, such as an "Electrotor."[4]

[2] Belcher, R., *ibid*. 1944, **30**, 280
[3] Stock, J. T., and Fill, M.A., *ibid*, 1950, **41**, 239-240
[4] Rev Motors, Ltd., Bolton, Lancs.

A Stand for Reactions on Microscope Slides

THE stand, which is constructed mainly of 5 mm. diameter glass rod is shown at (a) in Fig. 1. The central portion of the upper bar is of tubing of similar outside diameter to the rod. If a small hole A is blown in the tubing to act as an air release, the ends may be easily sealed to the rod to form a parallel-sided loop. Before sealing, the narrow strip of paper B, duly lettered or numbered, is inserted.
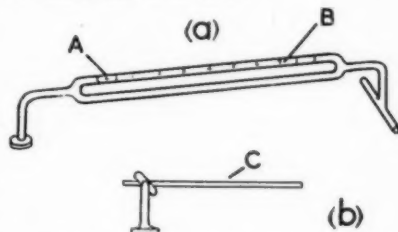

Fig. 1.—Stand for microscope slides.

The uprights are bent so that the plane of the loop makes an angle of about 45° with them, as shown at (b). The actual angle depends upon the inside width of the loop and the thickness of the microscope slides. It is found by trial so that, when one end of a slide C is inserted between the bars, the slide is supported horizontally and held by its own weight.

Society of Public Analysts
Microchemistry Group

THE Sixth Annual General Meeting of the Microchemistry Group was held at Sir John Cass College, London, E.C.3, on Friday, January 27th, 1950. It was reported that the number of members of the Group is now 309, an increase of 51 during 1949. The following officers and committee members were elected for the ensuing year : *Chairman*—Mr. Ronald Belcher. *Vice-Chairman*—Dr. Cecil L. Wilson. *Hon. Secretary*—Mr. Donald F. Phillips. *Hon. Treasurer*—Mr. Gerald Ingram. *Elected Committee Members*—Messrs. A. E. Heron, R. F. Milton, J. Sandilands, C. E. Spooner, D. W. Wilson, G. H. Wyatt.

An exhibition of new microchemical apparatus was organised by Dr. J. T. Stock, and a series of short papers describing the apparatus therein were read by Messrs. J. T. Stock, G. Ingram, W. T. Chambers, M. A. Fill, P. Heath, A. J. Lindsey, F. J. McMurray, W. Marshment, A. C. Mason.

The apparatus exhibited included the following :— (1) Split-type micro furnace. (2) One ml. syringe burette and stirrer outfit (described by Mr. Ingram). (3) Heating blocks for micro-electrolytic apparatus (Dr. Lindsey). (4) Combustion and absorption tube for the microdetermination of sulphur and halogens. (5) Simple pressure regulator for microcombustion trains (Dr. Chambers). (6) Semi-automatic microcombustion furnace (Mr. McMurray). (7) A photoelectric micro-nephelometer (Mr. Mason). (8) Micromagnetic bar stirrer. (9) Vacuum-operated stirring devices. (10) Clamps and stands for micro-apparatus. (11) Improvised microburette (Messrs. Fill and Stock). (12) Hydrogen sulphide delivery systems in semimicro qualitative apparatus (Messrs. Heath, Marshment and Stock). (13) Apparatus for conductometric micro-titration (Dr. Stock).

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Geiger Steel Analyser *

THE Geiger counter has been adapted by scientists of the Research Laboratory, United States Steel Corporation of Delaware, to the development of a quick and accurate method of analysing steel samples. After some of the problems that remain are solved, Geiger-counter analysis is expected to be even faster than the direct-reading spectrograph.

Just as it is capable of detecting and measuring radioactivity, this instrument is capable also of measuring invisible fluorescent rays, each of a special kind, that emanate from a steel sample when its atoms are ionized by powerful X-ray.

The sample's iron atoms and the atoms of alloying elements are not smashed in the analysis process, as might be expected. In fact, the steel sample is in no way harmed or changed.

Geiger-counter analysis is said to be simpler to make than an ordinary chemical or a spectrographic analysis of steel. Almost any piece of steel that has a flat surface will do for the purpose, so long as it is reasonably clean. Thus analysis might made of a random section of steel rail, the bottom of a cooking pot, a knife-blade, or a flattened mass of steel file dust.

A 20-milliamp 50-kv. molybdenum target X-ray tube is used in the laboratory studies. The ray floods the steel specimen, exciting secondary rays which are characteristic of the chemical elements alloyed with iron in the sample. These fluorescent rays are reflected through a narrow slit at right angles to the path of the primary X-ray to a crystal monochromator. This crystal is in the centre of a circle one fourth of whose circumference is traversed by the Geiger counter in its round of analysis. The counter is moved by motor in this scanning operation.

When used by a prospector for detecting radioactive substances in nature, the counter indicates their presence by clicks which can be counted accurately. The instrument used in the Kearny laboratory has a counting circuit, like other laboratory counters. In this case, however, the counter has been connected to a standard recording instrument, a self-balancing " potentiometer," which automatically plots the intensities of the various elements on graph paper.

This method of analysis is best adapted to detect and measure elements of " middle " atomic weight. Among these are the elements most often combined with iron and carbon to form the various steels in common use— chromium, manganese, molybdenum and nickel, as well as copper, which is frequently added to a steel composition to lessen corrosion. Also included in this class of alloying elements are some whose use is growing more and more frequent, such as niobium, titanium, tungsten and vanadium.

International Union of Crystallography

THE Executive Committee has accepted a kind invitation from the Swedish National Committee of Crystallography to hold the Second General Assembly and International Congress in Stockholm from June 27th to July 3rd, 1951. These dates have been chosen in consultation with the Swedish National Committee and with the National Committees of all the Adhering Bodies. It is hoped that this early notice will make it possible for many crystallographers to attend.

Spectrographic Methods Congress

THE 13th Congress of the Groupement pour l'Avancement des Methodes Spectrographiques will be held on June 21st to 23rd, 1950, at the Laboratoire Central de l'Armement, 1, Place St. Thomas d'Aquin, Paris, 7e. Four technical sessions are planned, with a visit to a works or laboratory on the afternoon of the 22nd. Anyone wishing to submit a communication to the Congress should send two copies of the typescript and illustrations to the Secretariat du G.A.M.S., at the above address.

Chemical Apparatus Exhibition and Convention

THE ACHEMA, an Exhibition covering the whole field of chemical apparatus, sponsored by the DECHEMA (Deutsche Gesellschaft für chemisches Apparatewesen) will be held in Frankfurt-on-Main during the week July 9th to 16th in conjunction with a meeting of that Society. The importance of the event for chemical science and technics, as well as for the design and construction of chemical apparatus will be appreciated. Some 350 leading firms engaged in the manufacture and distribution of chemical apparatus are exhibiting these products in six large halls, and efforts are being made to secure another hall to enable foreign manufacturers of chemical apparatus to take part in the Exhibition.

A whole series of new developments in the field of chemical apparatus is expected to be submitted for discussion at the meeting to be held in connection with this Exhibition. A large number of papers on the subject have been announced, and, during the Exhibition week the following meetings will be held : (1) General meeting of the Gesellschaft deutscher Chemiker ; (2) general meeting of the Deutsche Gesellschaft für chemisches Apparatewesen E.V. ; (3) special meeting of the VDI-Fachausschuss für Verfahrengs-technik ; (4) special meeting of the Sicherheitsingenieure der Berufsgenossenschaft der Chemischen Industrie ; (5) special meeting of the Verein Deutscher Eisenhüttenleute.

Invitations are being extended to those responsible for the instruction of chemical technics and the construction of apparatus and related subjects at technical colleges and universities in Germany and abroad to visit the ACHEMA IX with their students. Arrangements are being made to secure special railway and hotel terms. A special series of lectures for students is to be arranged.

Full details together with an invitation to attend are available on application to DECHEMA, Frankfurt am Main, Ulmanstrasse 10.

Operational Research Club

COMMENCING this month the Operational Research Club will issue a quarterly bulletin, whose field will be the application of the scientific method to human endeavour, both by itself and in relation to the use of materials and equipment.

Each issue will contain an important review article, by a leading authority in one of the various branches of the subject, together with informative abstracts specially prepared to bring out operational research methods and results in papers drawn from a wide selection, of countries, industries and fields of study.

Further particulars may be obtained from the Business Editor, Operational Research Quarterly, 25, Buckingham Gate, London, S.W.1.

Society of Instrument Technology
Formation of a Control Section

AUTOMATIC control mechanisms are not new, but during recent years such great strides have been made, accelerated by the need for extreme speed in gunnery, radar, and special requirements of new industrial processes, etc., that it is felt desirable to have some organisation devoted to encouraging progress in this subject. The Engineering Societies discuss some of its aspects on occasion, but the difficulty appears to be that the fundamentals of control are common to all branches of engineering and overflow into mathematics, physiology and psychology.

An attempt is now being made to bring together those who are interested in the scientific, technical and educational progress of the art.

In 1942, the Ministry of Supply Panel on Servo Mechanisms was formed as an informal body for the exchange of information. Its activities were organised by a small group who were only able to devote a small amount of time to the work. The range of subjects dealt with rapidly increased, and at each meeting a paper was read on a specific problem of design or application, the lecture being accompanied usually by a demonstration and followed by a discussion.

Although the Panel served a very useful purpose, it had not the necessary status to co-ordinate Service requirements and make recommendations for the allocation of research and development facilities, and for this purpose an Inter-departmental Committee was formed in 1944. The Panel survived, however, until the end of the war, and the Inter-departmental Committee was reorganised in 1946.

There are a number of activities which are not strictly speaking the business of the Inter-departmental Technical Committee on Servo-mechanisms, and many members of the old Servo Panel have regretted the absence of a forum where the scientific, technical and educational sides of the subject could be discussed freely. The Ministry of Supply recognises the desirability of such an association, and is anxious to help in its inauguration, although wishing to leave it free as soon as possible.

The Inter-departmental Committee has discussed this matter from time to time, and finally a Sub-Committee was formed, including several who were not members of the Committee, to represent the science as a whole so far as possible.

It was decided first of all to approach the Society of Instrument Technology, who, as their range of interests includes the subject, welcomed the idea of forming a Section which would appeal to all interests in the Control field. The Section is intended to cover the theory and practice of closed loop control systems, including Servo-mechanisms, Regulators and Process Controllers : also problems of the Human Operator as a Controller and when part of a larger Control system.

An inaugural meeting will be held on Tuesday, March 28th, at 6-30 for 7 p.m., at the Royal Society of Tropical Medicine and Hygiene, Mansion House, Portland Place, London, W.1, when three short papers will be read. These are as follows :—

Prof. K. A. Hayes—" Servomechanisms—Recent History and Basic Theory."

Dr. A. Utley—" The Problem of Stabilisation."

Prof. A. Tustin—" Problems in Control Systems which await Solution."

All who are interested in Control Mechanisms and allied subjects are invited to attend the inaugural meeting. If sufficient response is obtained, the inaugural meeting will be followed by a business meeting to deal with the formation of the Control Section within the framework of the Society of Instrument Technology

First International Microchemical Congress

THE First International Microchemical Congress will be held in Graz, Austria, from July 2nd to July 5th, 1950, under the auspices of the Austrian Society for Microchemistry. The provisional programme includes celebrations on the tenth and twentieth anniversaries of the death of Emich and Pregle, respectively, both of whom carried out their pioneering work on the development of microchemical techniques in Graz. There will be lectures by well-known microchemists covering the whole field of microchemistry, reports, exhibitions and receptions by Government and Municipal dignitaries. Honorary membership of the Osterreichische Gesellschaft für Mikrochemie will be bestowed on participants.

Forms of invitation may be obtained from the Hon. Secretary of the Microchemical Group of the Society of Public Analysts and other Analytical Chemists, Mr. D. F. Phillips, 10, Richmond Road, Blackpool, N.S., Lancs.

The Physical Society Exhibition

THE 34th Annual Exhibition of new developments in scientific instruments and apparatus will be held this year from March 31st to April 5th. The venue as last year will be the Imperial College, Imperial Institute Road, South Kensington, S.W.7.